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A STUDY OF THE "H" TEST FOR EVALUATING THE ADHESIVE PROPERTIES OF TIRE CORD IN NATURAL AND GR-S RUBBERS

By

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Southern Regional Research Laboratory
New Orleans, Louisiana

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STUDY OF THE "H" TEST FOR EVALUATING THE ADHESIVE PROPERTIES OF TIRE CORD IN NATURAL AND GR-S RUBBERS

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I. INTRODUCTION

The adhesion of cotton tire cord to natural (Hevea) rubber apparently never presented any special problem to the tire industry. However, the introduction of "high tenacity" rayon tire cords into tire manufacture became possible only after special adhesives had been developed[4] to bond the cord to the rubber, since the natural bond between synthetic fiber and rubber is very much less than for cotton. It has been frequently stated in the patent literature dealing with adhesives, that cotton cords contain numerous loose or projecting fiber ends which become imbedded in the soft rubber during vulcanization and thus form a strong mechanical bond in the resulting product. These fiber ends are not present in tire cords made from synthetic, continuous filaments and consequently, the relatively very weak bond between cords made of synthetic fibers and rubber was explained.

When the new GR-S rubber first came into production it was found that the stocks produced from it had an inferior adhesion to cotton tire cords as compared to that from natural rubber stocks. The inferior adhesion was especially pronounced at higher temperatures, and this was recognized as one of the more serious problems of the early synthetic tire program. Often separation would occur between cord fabric and rubber immediately after the tires came from the vulcanizing molds and before they had cooled. It was obvious that special attention would have to be given to this problem.

As a part of its contribution to the emergency war effort the Southern Regional Research Laboratory undertook a rather extensive study of cotton tire cord. The study of the adhesion of cotton cord to synthetic rubber was a logical extension of this study.

A first step in the study of adhesion seemed to be the selection or development of a suitable method for testing this property. The only published method that could be found was that of the American Society for

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1/ Present address, Research Laboratory, The Firestone Tire and Rubber Company, Akron 17, Ohio.

2/ Credit is due J. R. Jung, Jr., and Trinidad Mares for assistance with the experimental phases of this study.

3/ One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

4/ W. H. Charch and D. B. Maney, U. S. Patent 2,128,229, August 30, 1938. Assigned to E. I. duPont deNemours and Company.

Testing Materials[5], which involves the use of sections of finished tires, or fabric cured in rubber slabs. Obviously, the making of tires for a test of this type would be both inconvenient and expensive.

There were, also, in use by certain of the tire manufacturing and related firms, methods for the measurement of adhesion of tire cord to rubber. One such method, in use by The B. F. Goodrich Company[6] was said to give excellent evaluation of adhesion in rayon tire cords. This method employs cylinders of rubber 0.7 inch in diameter and one inch long, as described by Lessig[7] for the Goodrich flexometer, except that a section of the test cord passes diametrically through the center of the cylinder. Adhesion is measured by applying pressure to the ends of the cylinder at right angles to the cord axis, until the bond between the rubber and cord is broken, and noting the force required. The force measured is not the actual shearing force required to break the adhesive bond, but rather a functionally related compressive force.

Another adhesion method, in use in a number of commercial laboratories, was, and still is, the so-called "H" adhesion test. This test was originated and developed some years ago by the Goodyear Tire and Rubber Company for evaluation of the adhesion of rayon tire cords to rubber tire carcass stocks. Subsequently, around 1938, it was adopted by the Mansfield Tire and Rubber Company for the study of the adhesion of different constructions and gages of cotton tire cord[8]. Because of its more general use in a number of laboratories, and because of its previous application to cotton tire cord it was decided to make the initial study with the aid of this method.

Essentially, the "H" test may be said to be a measurement of the force required to pull a single cord, in the direction of its axis, from a strip of rubber in which one end of the cord over a given length is imbedded. Thus, the quantity measured is a shearing force acting across the cord-to-rubber interface. The opposite end of the piece of cord is held in the pulling clamp by lateral pressure from the jaws, transmitted through a second rubber strip. The two end-strips with the interconnecting cord form a specimen resembling the letter "H"; from this resemblance the test derives its name. The H-shaped specimens are obtained by cutting them from a cast, especially molded for the purpose, and consisting of a number of rubber strips interconnected at intervals by cords each of which extends through all the strips. The diagram of a typical mold in Figure 1 suggests the general form of the cast.

5/ A.S.T.M. standards on rubber products. A.S.T.M. Designation D 413-39. Standard methods of test for adhesion of vulcanized rubber (friction test). Prepared by Committee D-11 on Rubber Products.

6/ Kindly communicated by members of The B. F. Goodrich Company staff.

7/ Lessig, E. T., The Goodrich Flexometer. Ind. Eng. Chem., Anal. Ed. 9:582-588 (1937).

8/ Personal communication from W. R. Marsden, Bibb Manufacturing Company.

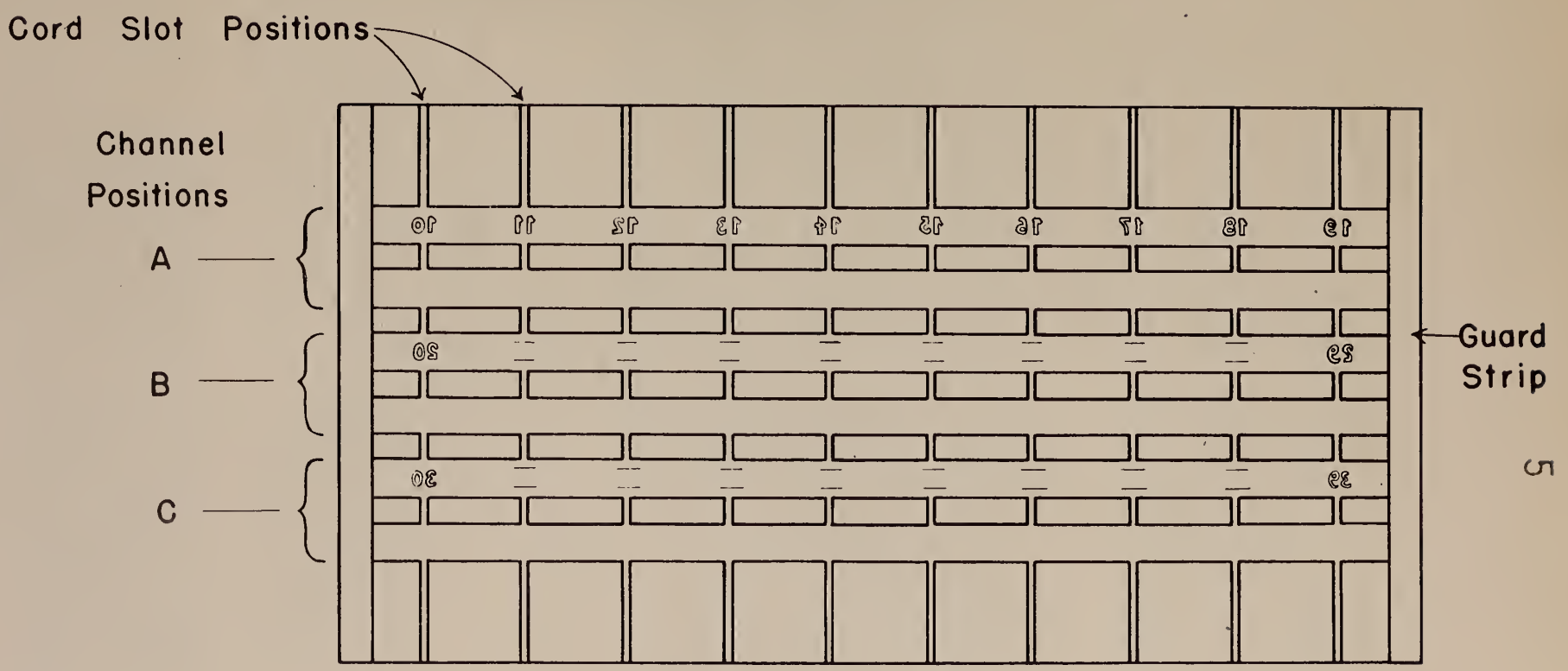

Figure 1. Armstrong Mold for "H" Adhesion Test.

Before undertaking a study of the "H" adhesion test on cotton tire cords a visit was paid to various commercial laboratories employing the test. It was learned that the technique was by no means standardized. Forming molds of widely different sizes and thicknesses were being used. One mold had a face 12 x 14 inches and a thickness of 3/4 inch. A second had a thickness of 5/8 inch and a face of 5-1/2 x 10 inches. Two other molds had thicknesses of approximately 1/4 inch and a face 6 x 6 inches. One mold had rubber strip channels 0.100 inch deep by 3/8 inch wide. A second mold had these channels the same width but 0.125 inch deep. One mold had numerals stamped at appropriate cord slot and channel positions so that each final test specimen would be individually numbered. Most molds did not have this provision.

The techniques employed in the different laboratories differed in a number of other particulars. In one case the uncured rubber stock was backed by "frictioned" or rubber-impregnated, square-woven cloth; in a second case by unfrictioned cloth. In one laboratory during cure a pressure of approximately 1800 p.s.i. was used on the face of the mold; in a second of approximately 550 p.s.i.; and in a third of only 110 p.s.i. In testing, clamps varying somewhat in general design and having jaw surfaces of distinctly different character were in use. Some had slots in both upper and lower jaws to straddle the cord; others only in the lower jaws. Speeds of the moving clamp of 2, 4, and 12 inches per minute were in routine use. From this it was evident that results from different laboratories could not be compared, and a selection of conditions must, of necessity, be made for suitable application. It is the purpose of this paper to present the experimental basis for the selections made, together with an outline of the proposed procedure.

II. EQUIPMENT AND MATERIALS

Molds, Press, and Testing Equipment

Two different molds were available for experimental use. Most of the castings containing the test specimens were made on a mold constructed from specifications received from the Armstrong Tire and Rubber Company. For convenience of reference this will be referred to hereafter as the "Armstrong" mold. The number and arrangement of the cord slots and rubber channels are shown in Figure 1. The plate is 5-1/2 x 10 inches x 5/8 inch thick.

The rubber strip channels are 3/8 inch wide by 0.100 inch deep and extend the full length of the plate to the guard strips at the ends. The channels are separated from each other by 1/4 inch and centered with respect to the width of the plate. The cord slots, of which there are ten, are 0.040 inch wide by 0.100 inch deep and displaced from each other by one inch on centers. The ends of the mold are equipped with guard strips which extend 1/2 inch above the face of the mold and between which a 1/2-inch steel cover plate fits.

A second mold was constructed according to specifications received from the Mansfield Tire and Rubber Company. This mold, which will hereafter be referred to as the "Mansfield" mold, is 12 x 14 inches over the face by 3/4 inch thick. In this mold the rubber channels and cord slots are laid out according to a pattern essentially the same as that of the Armstrong, except that the channels are 0.125 inch deep (instead of 0.100 inch) and only 8 inches long. The casting is located in the central portion of the mold, thus being surrounded by 3 to 4 inches of margin. The mold is covered with a 1/2-inch steel plate and recessed at the outer corners for insertion of the opening tool.

For curing, a hydraulic press, having steam-heated hot plates, approximately 19 x 20 inches, was used in most of the experiments. It was equipped with a 9-inch ram. The temperature was varied by controlling the steam pressure from a 120-pound service line, and read on a thermometer graduated in 2-degree intervals. Unless otherwise specified the cure was for 30 minutes at 287°F. in a previously heated mold, under a pressure of 550 p.s.i. on the mold face. The casting was then removed and allowed to stand for a period of time -- usually 24 hours or more.

The casting, prepared as indicated, was cut with shears or a paper cutter in such a way as to give one test specimen for each cord position and set of channel positions shown in Figure 1.

The adhesion of cord to rubber was measured in most cases on a Model L-6 Scott rubber-testing machine with spark recording, the capacity being set at 50 pounds. In some cases the reading was taken from the dial, in others from the chart. In the latter case the chart was first adjusted so that the lower zero position corresponded with the spark point when the pointer on the chart was reading zero and a spark recording was then made to indicate this base line. Subsequently a spark recording was made for each specimen at the maximum excursion of the pen after the cord had been stripped from the rubber. The platen carrying the chart was then moved down about 1/4 inch and the force required to separate the cord from the next specimen recorded. The zero was again checked at the conclusion of a series of measurements. In a few cases a Suter tire-cord tester with autographic recording was used, the capacity being set at 25 pounds.

The speed of the moving clamp (or "jaw speed" as it is frequently called) was 4 inches per minute in a number of the earlier tests and then 12 inches per minute, unless otherwise noted.

Special clamps were constructed for holding the specimens in the testing machine. These were modeled after those in use at the Mansfield Tire and Rubber Company, except that slots were used in only the lower jaws. However, clamps designed at the Armstrong Tire and Rubber Company were used in some tests. The upper clamp consists of a fixed and a hinged jaw both one inch wide, without slot. It is screwed onto the upper loading bar or chain. The jaws are closed with a wing-nut and provided with spring to spread the jaws when they are released. They have

a knurled surface. The lower clamp is similar in all respects except that it contains a slot 3/16 inch wide through the center of both jaws, is not provided with spring opening, and is not permanently attached to the machine. Some thirty of these were constructed, each provided with a hole to engage on a stud of an adapter on the lower loading bar. Thus, a number of specimens could be attached with one end in these lower clamps and all heated in an oven at a selected temperature. When they had reached the desired temperature the clamps with specimens could be removed, one at a time, and quickly slipped on the stud. The other end of the specimen could then be fixed in the upper jaw and the specimen loaded until separation occurred.

Since one of the objects of the study was to determine how the cord-to-rubber adhesion varies with temperature, usually half of the specimens from each casting were tested at elevated temperature. A temperature of 275°F. was used unless otherwise noted for the elevated temperature, the temperature of the room being about 75°F. The elevated temperature was, for all practical purposes, sufficiently near the maximum temperature at which a tire will fail rapidly in service. Fifteen specimens already fixed in lower clamps, as described above, were brought to this temperature by heating one and one-half hours in an electrically heated and controlled air oven having a precision of about one degree. Also, tests were made in which the temperature was varied systematically over a wide range.

Rubber Stocks

A total of five different tire carcass stocks[9/] were used in the present studies. The sample numbers, types of rubber, dates of receipt, and molds for which intended were as follows:

Co-767, natural, Sept. 10, 1943, Armstrong mold;
Co-768, GR-S, S-4, Sept. 13, 1943, Mansfield mold;
Co-783, GR-S, S-4, Sept. 21, 1943, Armstrong mold;
Co-1063, GR-S, S-4, Nov. 20, 1943, Armstrong mold;
Co-1352, GR-S, S-4, Feb. 29, 1944, Armstrong mold.

The stocks intended for use in the Armstrong mold had been sheeted, rolled onto frictioned, square-woven backing cloth to give a gage of about 0.055 inch, and then covered with Holland cloth. The proper vulcanization time for these stocks, both natural and GR-S, was given as 30 minutes at 287°F. The Mansfield stock, Co-768, was sheeted to a gage of approximately 0.070 inch and then rolled onto a plain 8.60-ounce 2-ply unfrictioned duck to give an over-all gage of 0.105 inch. The other surface was covered with Holland cloth.

9/ These stocks were kindly furnished by the Armstrong Tire and Rubber Company of Natchez, Mississippi, or, in one case, by the Mansfield Tire and Rubber Company of Mansfield, Ohio.

Tire Cords

For most of the measurements two different tire cords, both manufactured from the same Stoneville 2B cotton, were employed:

1. One cord, designated Co-752, was of 2/3/4/3 construction. It had a cable twist of 10.5 turns per inch and a gage of approximately 0.0285 inch. For convenience this cord will be referred to hereafter as "low gage".

2. The other cord, designated Co-753, was of 23/5/3 construction. It had a cable twist of 8.7 turns per inch and a gage of approximately 0.0315 inch. It will be referred to hereafter as "medium gage".

Certain other tire cords were used in special studies but these will be described in connection with their particular applications.

III. INFLUENCE OF EXPERIMENTAL CONDITIONS ON OBSERVATIONS

Pre-drying of the Cord

In order to determine whether the moisture normally present in tire cord as it stands in the laboratory (4.0 to 6.0 percent) influences adhesion, two experiments were carried out. In the first one, short sections of the low gage tire cord, Co-752, were prepared and one-half placed in an open-mouthed bottle in an oven at 220°F. After 2-1/2 hours the bottle was quickly stoppered and allowed to cool. Strips of natural rubber carcass stock, Co-767, were laid face up in the channels of the previously heated mold, and then undried and the above-described oven-dried cords were laid alternately in the cord slots across the mold. Additional strips of the natural rubber stock were now laid face down over the cords in the channels and the cover was laid on. All of this was done as expeditiously as possible and the maximum time of exposure of the dried cord to the atmosphere was perhaps four minutes. After cure and standing 120 hours the resulting specimens were tested at room temperature, a jaw speed of 4 inches per minute being used. The means (of fifteen observations each) of the force required to separate the cord from the rubber were: for the undried cord, 13.98 $\pm$ 0.99 pounds[10/], and for the dried cord, 13.47 $\pm$ 1.13 pounds. These data indicate that the pre-drying of cotton cord produces no significant effect on adhesion to natural rubber carcass stock at room temperature.

10/ As in all tables throughout this report, the quantity appended to each mean value is the standard error, calculated by means of the familiar relation $S_{\bar{x}} = \sqrt{\sum d^2/n(n-1)}$,
where d = deviation of an individual observation from the mean, and
n = number of observations in the test, 10 unless otherwise indicated.

A second somewhat more comprehensive test was carried out, using synthetic GR-S carcass stock, Co-783, and testing the pieces at both room temperature and at 275°F. The same cord, Co-752, was used as in the preceding experiment and dried in the same way. The results, also at a jaw-speed of 4 inches per minute, are summarized in Table 1.

Table 1. Force Required at Two Temperatures to Separate Normal and Dried Tire Cord from GR-S Carcass Stock.

Condition of cord	Testing temperature	Force: Mean	Force: Coefficient of variation
	°F.	Lbs.	Percent
Undried	75	14.57 ± .19	4.80
Dried	75	14.13 ± .21	5.64
Undried	275	6.49 ± .19	11.47
Dried	275	6.05 ± .16	10.05

The foregoing results show a slight tendency for the adhesion of the undried cord to be greater than that of the dried cord. However, when an analysis of the variance was made, the probability that the drying treatment did influence the results was found to be small. Surprisingly enough the trend is toward larger adhesion forces in the cord which was not dried, whereas the contrary trend would be expected on the basis of the known antagonism between rubber and moisture. In order to learn whether there was any difference in the uniformity of the results with or without drying, the coefficients of variation were computed for each test. These are presented in the fourth column of Table 1. They certainly do not suggest any particularly advantageous effect of drying on the uniformity of results. In view of these tests it was concluded that the drying of untreated tire cord before use in the adhesion test could be safely dispensed with under ordinary circumstances. In the case of treated tire cord the influence of moisture on the adhesive itself would naturally dictate the procedure to be followed.

Pressure on Mold during Cure

Since the various laboratories employing the "H" adhesion test were applying such widely differing pressures on the molds during cure, experiments were undertaken to determine the influence of pressure. In an overfull adhesion mold during cure under pressure it is obvious that it will not be possible to build up a pressure on the rubber stock itself equal to the pressure on the mold, since the rubber becomes quite fluid, at least during the early stages of cure, and is free to move out through the open cord slots. Consequently, it is not clear why more than a minimum pressure on the mold, sufficient to cause flow into the surface of the cords, should have influence on the results.

Two experiments were conducted. In the first, the natural rubber carcass stock, Co-767, was employed and the pressure was varied through a comparatively narrow range of four levels from 450 to 615 p.s.i., calculated on the area of the mold face. The low-gage cord, Co-752, was used. The rubber strips and cords were inserted and the mold, under the selected pressure, heated for 30 minutes at 287°F. The resulting cures were removed from the mold, allowed to stand for 24 hours or longer, and specimens tested both hot and cold, at a jaw-speed of 4 inches per minute. The results are presented in Table 2, each value being the mean of ten observations.

Table 2. Force Required at Two Temperatures to Separate Cotton Tire Cord from Natural Rubber Carcass Stock, Cured at Different Pressures.

Pressure during cure	Tested at 75°F.	Tested at 275°F.
p.s.i.	Lbs.	Lbs.
456	13.55 ± 0.41	5.68 ± 0.41
502	13.37 ± 0.33	4.78 ± 0.31
570	14.07 ± 0.46	6.91 ± 0.25
616	13.45 ± 0.39	5.53 ± 0.33

Examination of the data in Table 2 shows considerable variation in results but no consistent trend with the pressure used during cure, regardless of whether the specimens are hot or cold when tested. It may be pointed out that variations of pressure in the present test were associated, by necessity, with separate cures and that the differences found could have been due equally to slight variations in temperature or time of cure or even to other causes. It is highly significant that in Table 2 there is no consistent trend of adhesive forces with pressure during cure so that it must be concluded that other factors are influential and disturb the results.

In the second experiment on the influence of pressure on the mold during cure GR-S carcass stock, Co-783, was used and the pressure was varied over a much greater range. Furthermore, in this experiment two tire cords, Co-752 and Co-753, having different gages were employed. Cords having the two gages were laid alternately in the cord slots across the mold. The results, obtained at a jaw-speed of 4 inches per minute, are recorded in Table 3.

Analysis of variance was applied, separately, to the data obtained at the two different temperatures. The results indicated that pressure during cure has no significant influence on the results of cold measurements but suggested that it does influence the results when the adhesion is measured hot. Examination of Table 3 shows that most of the latter difference is associated with the lowest pressure and a more detailed statistical test showed that the difference between means for the 290-

and 542-p.s.i. cures was not significant. Tire-cord variables (gage, twist, construction) influenced adhesion very significantly at room temperature but, at high temperature, while the trend was consistent the sample variation was so large as to overshadow this.

Table 3. Force Required to Separate Cotton Tire Cords of Two Different Gages at Two Temperatures from Synthetic GR-S Rubber Carcass Stock, Cured at Different Pressures.

Pressure during cure	Tested at 75°F. Low gage	75°F. Medium gage	75°F. Mean	Tested at 275°F. Low gage	275°F. Medium gage	275°F. Mean
p.s.i.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.
27.5	13.37±0.36	14.83±.45	14.10	5.46±.26	5.92±.29	5.69
290	13.59±.33	15.54±.41	14.56	6.22±.19	6.55±.29	6.39
542	13.57±.32	15.16±.24	14.36	6.44±.22	6.85±.31	6.64

The results of the foregoing experiments have been summarized in Figure 2. By reference to this figure it will be observed how slight are the effects of curing at different pressures. In routine work it would seem advisable to use a pressure during cure which is of a similar order of magnitude to that used in tire manufacture, viz. 250 p.s.i., with the knowledge that the mold itself probably supports the greater part of this.

Testing Temperature

That the adhesion of cords to rubber varies greatly with the temperature at which measurements are made has already been mentioned. As Tables 1, 2, and 3 clearly reveal, there is a major decrease in the adhesive force when the testing temperature is raised from 75° to 275°F. Analyses of the variance in the data summarized in Tables 1 and 2 indicate that testing temperature exerts a far greater influence on the adhesive force than either of the other factors.

In order to obtain fuller information on the behavior of cord-to-rubber adhesion as a function of temperature, a series of tests employing four different rubber stocks, and covering a range of temperatures from that of the room (77°F.) up to 300°F., were undertaken. The four rubber stocks used included the one of natural rubber, Co-767, and three of the GR-S stocks previously mentioned. The cotton tire cord used throughout the tests was the medium-gage sample, Co-753, and the jaw speed was 12 inches per minute.

The results are shown graphically in Figure 3. Consistently, all curves show an initial drop as the temperature rises to about 160°F. Between this temperature and about 250°F. the rate of decline is small; above 250°F. the rate is again accelerated. It can be seen from the

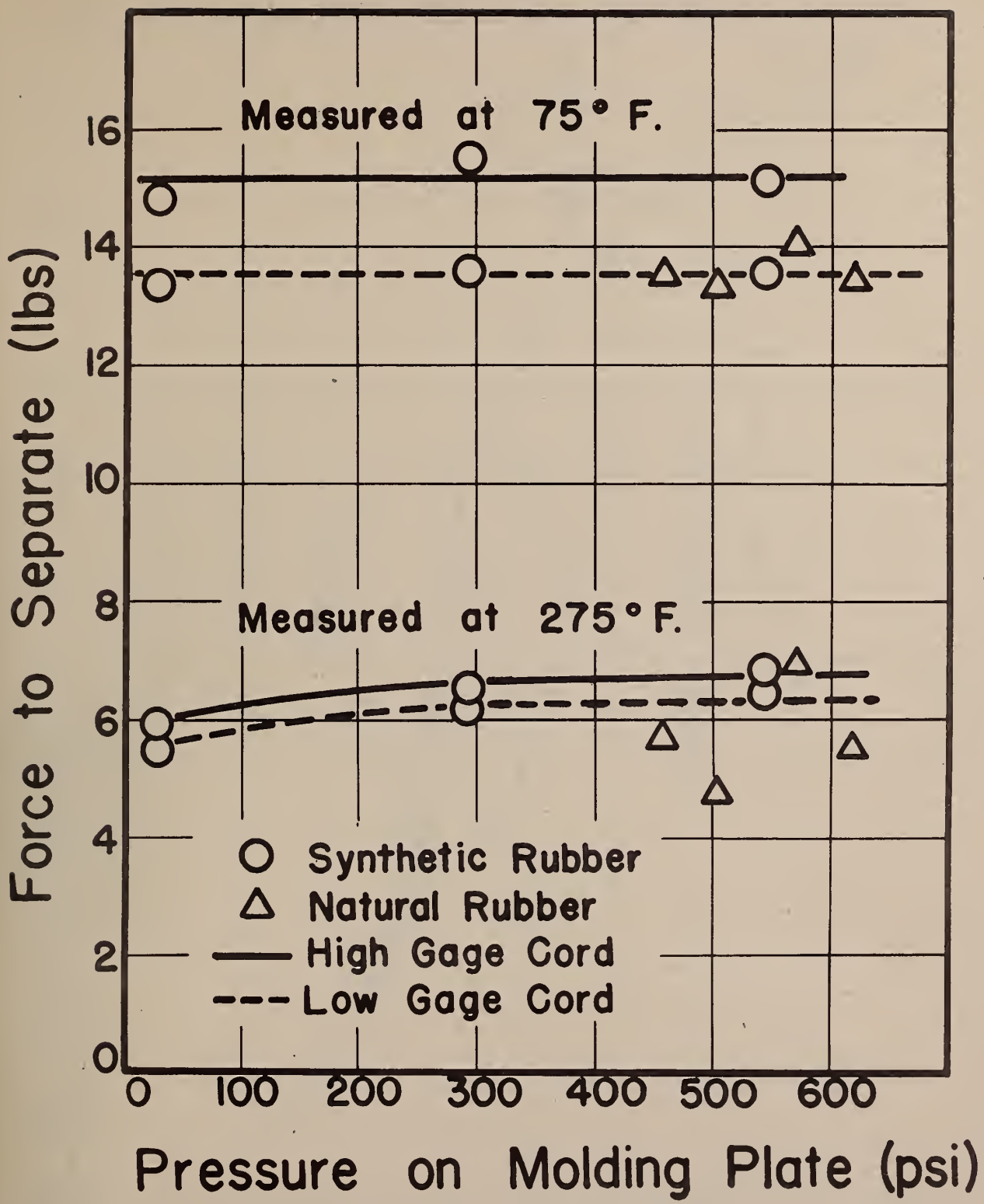

Figure 2. Effect of Pressure, Gage, and Temperature on Rubber-Tire Cord Adhesion.

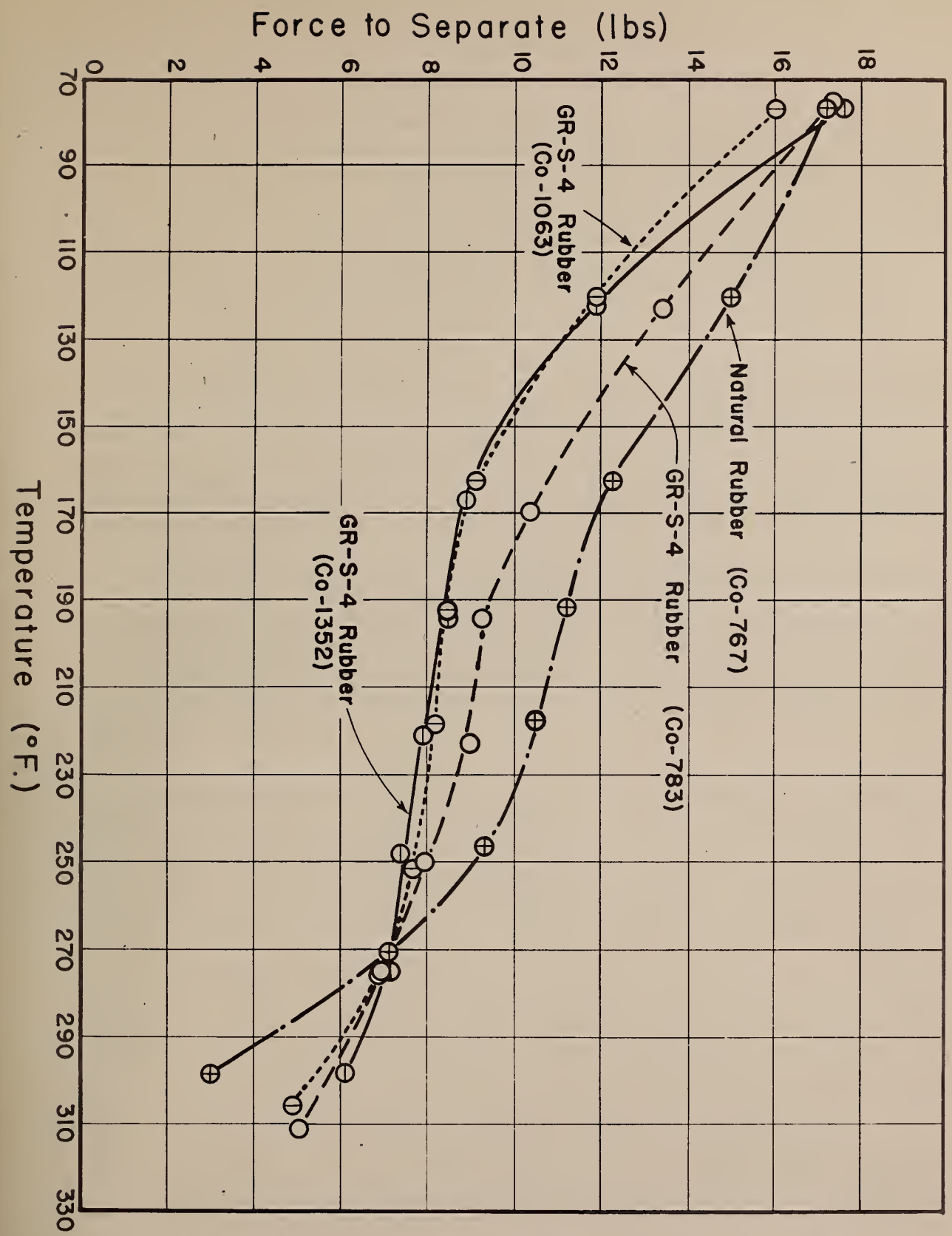

Figure 3. Adhesion of Various Rubber Stocks to Cotton Tire Cord at Different Temperatures.

curves that the adhesion of this cord at room temperature and again at about 275°F. is the same for all four stocks. Between these points, natural rubber exhibits the best adhesion, but above 275°F. the adhesion of natural rubber falls off more rapidly than that of the synthetic stocks. The difference in behavior here between natural and GR-S rubber stocks appears to conform to the theory of a lower thermal stability in the junctures of the natural rubber network, since researches by Tobolsky, Prettyman, and Dillon[11] suggest that at 212°F. and above, the intermolecular bonds in natural rubber are more susceptible to rupture than are those in GR-S stock.

In the neighborhood of 275°F. the adhesion is not excessively sensitive to temperature changes and is quite uniform among the various rubber stocks. A temperature of 275°F. appears to provide about the highest temperature to which the adhesion specimens can be exposed without severe damage to the adhesive properties. For these reasons 275°F. appears to be well recommended as a routine temperature for use to determine the adhesion at high temperature. Undoubtedly, special occasions will arise where adhesion measurements will be required at various other temperatures, also.

The question was raised as to whether high-temperature measurements of adhesion were needed at all, and whether the high-temperature adhesion behavior could not be predicted from room temperature measurements. The foregoing discussion of Figure 3 shows that, even though the adhesions at room temperature are all of nearly the same magnitude, different rubber stocks have different adhesions at higher temperatures. Furthermore, the adhesion-temperature relationship is not linear, nor are the curves parallel.

It was decided to determine the correlation coefficient between the adhesion measured at 75°F. and at 275°F. on a series of tire cords, and thus demonstrate how close the relationship is. For this purpose, 55 pairs of adhesion values obtained at the two temperatures were tabulated for cotton, rayon, and mercerized-cotton tire cords. Some of these were untreated, and others had been treated with both commercial and special adhesive compounds to improve the adhesion of the cord to GR-S rubber. Before computing the correlation coefficient, the adhesions were expressed as "characteristic adhesion" and also adjusted to the mean of the "basic control" as will be described in a following section. These adjustments removed the effect of varying gage of the cord, as well as the effects of uncontrolled variations from test to test. The resulting correlation coefficient, thus obtained, was 0.88, giving a coefficient of determination of 0.775. The latter signifies that only 77.5 percent or slightly over three-fourths of the variability in the adhesion at 275°F., can be predicted from a measurement of the adhesion at 75°F. The standard error of estimate amounted to 10 percent. In view of this rather poor prediction, it is recommended that "hot" adhesion measurements be made as a routine part of the testing procedure.

11/ A. V. Tobolsky, I. B. Prettyman, and J. H. Dillon, J. App. Phys. 15, 380, (1944).

Speed of Pulling Clamp

In the early experimentation a clamp speed of 4 inches per minute was arbitrarily used. In order to learn how variation in speed of the pulling clamp of the testing machine might influence the results, three cures were conducted using the medium-gage cord, Co-753, and GR-S rubber carcass stock, Co-783. The positions of the test pieces in the mold were identified according to the letter and numeral scheme shown in Figure 1, and selected for clamp speed variation in such a way that all mold positions were employed for each speed in both hot and cold adhesion measurements. The mold positions and adhesion forces for each clamp speed and temperature are shown in Table 4, each tabulated value referring to a particular specimen.

By reference to Table 4 it is at once evident that clamp speed quite definitely influences the adhesion forces both when tested at room temperature and when tested at elevated temperature. With increasing clamp speed the values increase progressively. Analysis of variance, which also includes channel and cord positions in the mold, was applied with the following results:

Source of variance	Degrees freedom	Sum of squares	Mean square	Value of F. Found	5%	1%
Test temperature (1)	1	1784.008	1784.008	4784.15	-	7.01
Clamp speed (2)	2	78.433	39.216	105.18	-	4.92
Channel position (3)	2	11.865	5.932	15.93	-	4.92
Cord position (4)	4	1.103	.276	-	-	-
Interaction (1)vs(2)	2	9.481	4.740	12.71	-	4.92
" (1)vs(3)	2	2.015	1.008	2.70	3.13	-
" (2)vs(3)	4	2.329	0.582	1.56	2.50	-
Error	72	26.843	0.3728			
Total	89	1916.077				

Separate analyses of variance of the data at the two testing temperatures were also made and disclosed a significant difference in sampling error. However, since the separate consideration of the data made no difference in the conclusions except for the single case of the interaction of clamp speed and channel position which was shown to be significant at the higher temperature, the difference in sampling errors was neglected and the data for the two temperatures pooled for analysis.

It is evident that the variance due to clamp speed is highly significant. The relation at the two testing temperatures is shown in Figure 4. It is not surprising to find differences in the adhesion forces depending on clamp speed, since this long has been common observation in tensile testing. Explanations for the use of speeds lower than the more conventional ones have been that it was desired to increase the load gently during test and not jerk the cord out of the rubber. The actual situation seems to be that the adhesion separation is a plastic phenomenon involving a relaxation time.

Table 4. Force Required at Three Clamp Speeds and Two Temperatures to Separate Cotton Tire Cord from Synthetic GR-S Carcass Stock.

Measured at 75°F.						Measured at 275°F.					
2 in./min.		4 in./min.		12 in./min.		2 in./min.		4 in./min.		12 in./min.	
	Lbs.		Lbs.		Lbs.		Lbs.		Lbs.		Lbs.
A_1	14.7	A_1	14.7	A_1	16.5	A_{10}	6.4	A_{10}	6.4	A_{10}	8.0
A_3	13.4	A_3	15.4	A_3	16.9	A_8	6.3	A_8	5.5	A_8	7.7
A_5	13.7	A_5	14.9	A_5	18.4	A_6	6.3	A_6	6.1	A_6	8.0
A_7	13.1	A_7	14.9	A_7	17.0	A_4	6.7	A_4	5.8	A_4	7.7
A_9	13.2	A_9	15.8	A_9	16.6	A_2	6.4	A_2	6.1	A_2	7.8
B_1	14.9	B_1	15.2	B_1	16.7	B_{10}	6.4	B_{10}	6.6	B_{10}	7.6
B_3	13.6	B_3	15.5	B_3	16.4	B_8	5.8	B_8	7.5	B_8	7.7
B_5	14.6	B_5	16.4	B_5	16.7	B_6	5.9	B_6	6.4	B_6	7.8
B_7	14.2	B_7	15.6[1]	B_7	17.8	B_4	6.4	B_4	6.7	B_4	7.4
B_9	14.1	B_9	15.4	B_9	18.1	B_2	5.9	B_2	6.4	B_2	7.0
C_1	13.6	C_1	15.2	C_1	17.5	C_{10}	6.4	C_{10}	7.4	C_{10}	8.2
C_3	14.8	C_3	16.8	C_3	18.4	C_8	6.7	C_8	6.7	C_8	8.2
C_5	16.7	C_5	16.4	C_5	17.5	C_6	7.0	C_6	7.4	C_6	8.0
C_7	14.8	C_7	17.0	C_7	17.5	C_4	6.4	C_4	7.2	C_4	8.0
C_9	16.5	C_9	15.2	C_9	19.6	C_2	5.7	C_2	7.7	C_2	7.5
Av.	14.39		15.63		17.44		6.31		6.66		7.77
Av. 2 in./min., 10.36			Av. 4 in./min., 11.14				Av. 12 in./min., 12.61				

1/ Specimen broke at 1.5 lbs.; mean value of column used for variance analysis.

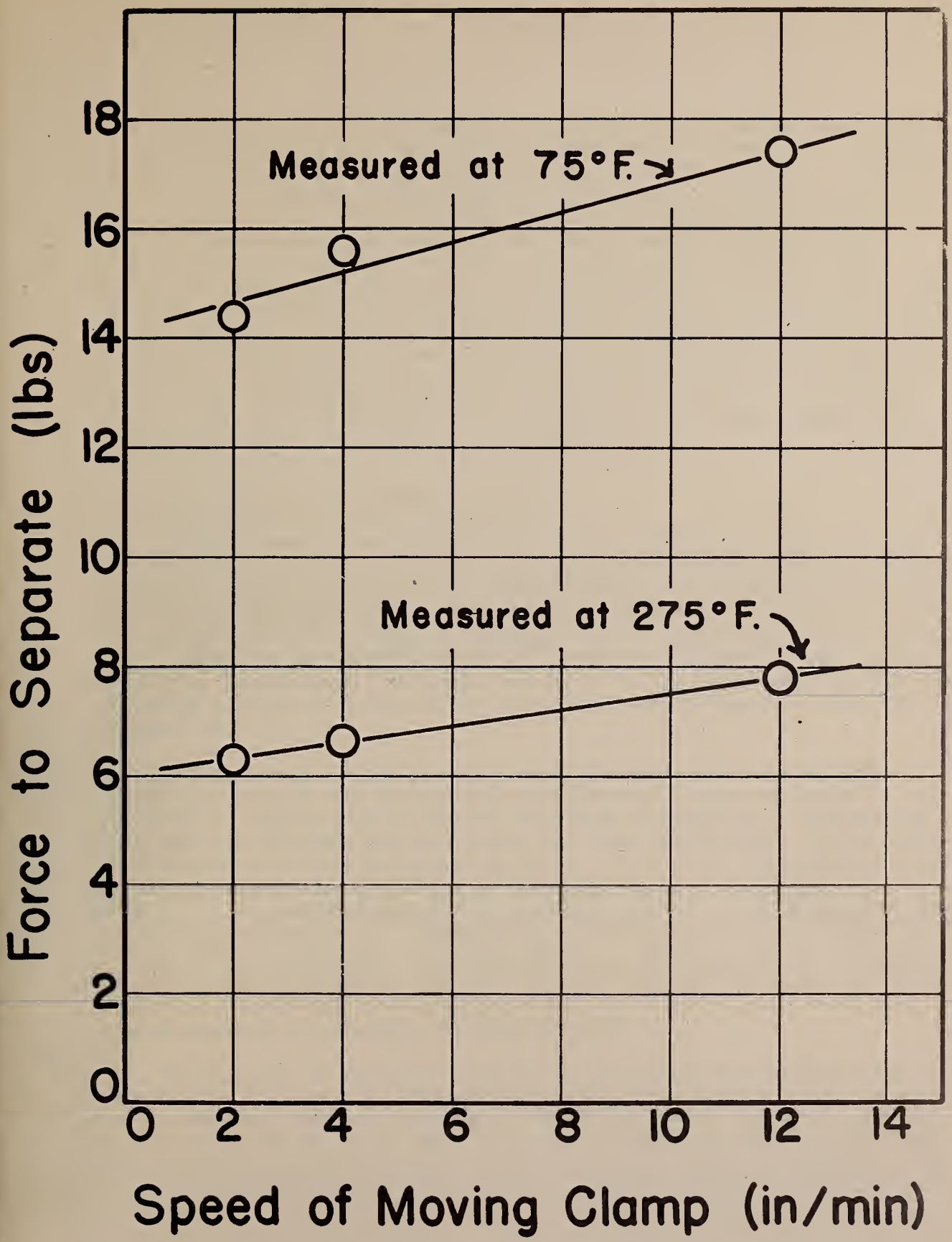

Figure 4. Effect of Clamp Speed on Rubber-Tire Cord Adhesion.

There are several advantages to be gained from using the higher speeds of moving clamp. First, the adhesion values usually obtained are rather low for conventional textile testing machines such as those of 25-50 pound capacity. Increasing the magnitude of the reading will bring it into a more favorable range, and will further improve the test by reducing the relative importance of small frictional losses and the like. Second, a speed of 12 inches per minute is more conventional in textile testing machines of small capacity, such as are employed not only in the tire-cord manufacturing industry but in the tire manufacturing industry as well, and the use of this conventional clamp-speed will make the test more convenient of adoption, generally. Third, the higher speed of separation would seem to be in the right direction to approach more nearly the rate of loading of the cord in a tire in actual use, although of course it is still far short of it. For these reasons it is believed that a speed of 12 inches per minute should be used for the moving clamp.

Position of Test Piece in the Curing Mold

From the start there has been some opinion that the relative location or position of the adhesion specimens in the mold during cure might have an influence on the adhesion values finally obtained. This would be due to temperature gradients between the outer edge of the mold and the interior. The finding of some influence apparently associated with the cure, in tests thought to be otherwise entirely comparable, tended to augment this suspicion. Accordingly, advantage was taken in the study presented in the preceding Section to obtain at the same time some information along the present line.

As pointed out in the previous topic the mold positions had been mapped according to the channel and slot positions shown in Figure 1. By reference to Figure 1 it is evident that each combination of channel and cord position provides one test piece for final measurement. It is likewise evident that cord positions 10, 9, 8, 7, and 6 are respectively equivalent, in relation to the center of the mold, to cord positions 1, 2, 3, 4, and 5. Furthermore, channel positions A and C are equivalent, relative to the sides of the mold. However, channel position B is central and has no comparable counterpart. For this reason, in the analysis of variance presented in the preceding Section, the three channel positions were taken separately, while the cord positions 1 and 10, 2 and 9, etc., were used interchangeably in arranging the data of Table 4.

The analysis of variance shown in the preceding Section indicates a significant difference in adhesion associated with channel positions but none associated with cord positions. This result is somewhat paradoxical since, if differences of adhesion existed, it might have been supposed that cord positions would more likely show them than channel positions. A more detailed examination of the data shows the mean adhesion for channel A to be 11.01, that for channel B, 11.22, and that for channel C, 11.87. Thus, the average adhesion for channels A and C which were symmetrical and similar with respect to the mold map gave mean adhesions which were smaller and larger, respectively, than that of the central channel B. Thus, the

conclusion must be reached that any source of influence of channel positions on adhesion must have been exterior to the mold -- perhaps a gradient of temperature on the platens of the press or slight damage in removing the casting from the mold, or even a gradient in the rubber stock composition. Since three different cures were employed in the present analysis the possibility existed that these cures might have introduced a variable which was not taken into account in the analysis used. A separate examination of the variance due to cure, therefore, was made but this was found to show no significant effect. These data seem to indicate therefore that there is no serious influence of mold positions under the test conditions employed. Nevertheless, in all tests recorded here it has been the practice in any one test to lay the series of test cords in the cord slots in a progressive succession in such a way that each cord sample of the test occupies as many different cord slots in the different cures as possible.

Character of Jaw Surfaces and Pressure on Clamps

A tentative opinion has prevailed in some quarters that the measurement of adhesion in the "H" test is quite sensitive to the conditions under which the test specimens are attached in the jaws of the tester clamps. Thus, different clamping surfaces, found on different jaws used to support the adhesion specimens, have been supposed to introduce differences into the results. Accordingly, comparison was made of adhesion-values obtained by the use of two clamps having markedly different jaw surfaces.

One type of clamp, employed in the laboratory of the Armstrong Tire and Rubber Company, has deep serrations, or V-shaped grooves, about 3/16 inch wide, running across the width of the jaws on each side of the slot. The grooves are on both jaws and are positioned so that they intermesh when the jaws are closed. The tendency, thus, is to produce corrugations in the rubber support strips of the specimens during measurement. The other type of clamp, used most frequently in the present study, has knurled jaw surfaces, with grooves less than 1/64 inch deep.

Adhesion-specimens, prepared in the usual manner from natural rubber stock Co-767 and employing two different, treated tire cords, were tested with the two types of clamps. The clamp used in the upper position (obviously having no influence on the measurements) remained the same throughout the tests. The testing was one with a jaw speed of 4 inches per minute. Results are given in Table 5.

The apparent increase in the mean adhesion-values in Table 5 from the use of jaws with knurled surfaces was found on statistical analysis, to be entirely without significance, at least in tests at room temperature. Because of the increased fluidity of rubber at 275°F. we may infer that the character of the clamping surface would be even less likely to influence adhesion measurements than at 75°F. It appears that any corrugations which may be put in the rubber support strip of the test specimen by the serrated jaw surfaces, because of the softness of the rubber, are not

communicated across the jaw slit to the imbedded cord, and thus do not affect the adhesion measurements.

Table 5. Force Required at 75°F. to Separate Two Treated Cotton Tire Cords from Natural Rubber, when Supported in Clamps Having Different Types of Jaw Surfaces.

Cord sample	Serrated jaw	Knurled jaw
	Lbs.	Lbs.
I	11.95 ± .62	12.52 ± .48
II	8.42 ± .37	9.35 ± .85

The pressure with which the test specimen is clamped has been advanced as another condition to which adhesion values may be more or less sensitive. However, it may be concluded that, as the rather significant deformation of the rubber support strip of the specimen by the serrated jaws is not reflected in the measurements, neither are differences in clamping pressure on the lower jaw (the one with the slit). The conditions under which the upper jaw holds the test specimen are obviously not important from any aspect.

Calendering-Direction in Rubber Channel Strips

The mechanical anisotropy of rubber, resulting from calendering operations, is well known. It was therefore reasonable to admit the possibility that this anisotropy might be reflected in a difference in adhesion-values between "H" type specimens in which the cord is imbedded parallel to the calendering direction and those in which the cord is perpendicular to that direction. Furthermore, it is possible that the directions of the warp and filler of the backing cloth with respect to specimen construction might lead to differences. Evidently if such differences existed and were significant, care would have to be exercised in cutting the skim-stock strips so that in all cures they would be oriented uniformly with respect to calendering and backing cloth directions.

To investigate this effect test specimens were molded with the imbedded cord in the direction of calendering, i.e., warpwise, in some specimens, and perpendicular to this direction in others. The natural rubber carcass stock, Co-767, and the low-gage cord, Co-752, were used in this experiment. The results of the measurements at two temperatures are given in Table 6.

Table 6. Force Required at Two Different Temperatures to Separate Cotton Tire Cord Imbedded in Natural Rubber Parallel and Perpendicular to Direction of Calendering, from the Rubber

Testing temperature	Cords parallel to calendering direction	Cords perpendicular to calendering direction
°F.	Lbs.	Lbs.
75	11.93 ± .18	11.66 ± .21
275	4.58 ± .13	4.41 ± .25

Aside from reflecting the well-known preponderant effect of testing temperature on adhesion values, these data indicate definitely that the orientation brought about in the test specimen by the calendering operation has no influence on the adhesion values. At the same time these results indicate that there is no distinction, insofar as adhesion measurements are concerned, between warp and filler in the backing cloth on the rubber, since the warp and filler directions of the yarns were necessarily interchanged when the calendering direction of the rubber strip was altered in these tests. While these results specifically apply only to natural rubber stock, the absence of any orientation influence in this material, which is known to be susceptible, is so clear-cut that the influence of strip direction on other stocks seems very unlikely.

Differences in Molds

Adhesion values may reflect differences in linear dimensions of the mold, depth of channel, etc., through the possibility that the temperature-gradients induced by different mold dimensions will influence differently the curing of the specimens and hence the adhesion.

The results of three tests employing the medium- and low-gage tire-cord samples have been combined to get a representative example of the influence of differences in molds and rubber stock on adhesion values. Because the two molds used have channels of different depths, they require rubber stocks of different gages. This requirement implies that differently sheeted rubber must necessarily be used in each mold, so that it is not possible to segregate completely the effects of different molds from that of different rubber sheets. While it is possible to produce two or more rubber sheets having different gages but a uniform composition, such sheets were not procured for the present experiment.

The two molds and two rubber stocks employed in this test are described under "Equipment and Materials". The Armstrong and Mansfield molds were used and the two rubber stocks were type S-4 GR-S carcass stocks, Co-783 and Co-768. Throughout this experiment the loads on the hydraulic press were adjusted so that the curing pressure was always

250 p.s.i. on the mold face. The manufacturers' instructions indicated cures of 30 minutes, at 287°F. for Co-783 and at 260°F. for Co-768. However, in the latter case an obvious undercure resulted and the casting had to be discarded. Since a second cure had already been started at 260°F. and the stock was nearly exhausted, the time was extended to 78 minutes, which according to the table published by the Office of Rubber Director[12] should be equivalent to 287°F. for 30 minutes. The same stock used on the Mansfield mold was cured 30 minutes at 287°F. The high gage of this stock resulted in a heavy flash in the Armstrong mold. Unfortunately, the gage of stock Co-783 was too low to permit its use in the Mansfield mold. The data obtained at two testing temperatures are presented in Table 7.

Table 7. Force Required to Separate Cotton Medium- and Low-Gage Tire Cords from Two Different GR-S Synthetic Carcass Stocks Cured in Two Different Molds.

Testing temperature	Cord gage	Armstrong mold: Stock Co-783	Armstrong mold: Stock Co-768	Mansfield mold and stock Co-768	Average force
°F.		Lbs.	Lbs.	Lbs.	Lbs.
75	Medium	17.81±.28	15.64±.26	14.14 ± .23	15.86
75	Low	15.47±.29	15.06±.30	12.96 ± .22	14.50
275	Medium	8.04±.18	6.96±.40	6.17 ± .11	7.06
275	Low	6.86±.29	5.98±.62	5.10 ± .15	5.98

The data in Table 7 show for both the medium- and low-gage cord the highest adhesive forces from stock Co-783 used with the Armstrong mold. Stock Co-768 gave somewhat lower adhesion when cured in the Armstrong mold, and still lower values when cured in the Mansfield mold. The trends are the same at both testing temperatures. Since there were considerable variations in the individual observations obtained under the different conditions, analyses of variance were made separately for the measurements at the two temperatures. These analyses indicated that the use of different molds, stocks, and cures resulted in highly significant differences in the forces required to strip the cord from the rubber. The differences in adhesive values between the two rubber stocks are discussed in Sec. V, "Rubber-Stock Factors Influencing Adhesion".

On comparing the results with rubber stock Co-768 in the two molds, it was found that the difference in adhesion values measured at 75°F. is probably statistically significant where the low-gage cord is used but not in any other case. It should be pointed out that although the stock was the same for both molds the cures were not identical, being 78 minutes at 260°F. for the Armstrong mold and 30 minutes at 287°F. for the Mansfield mold.

12/ Office of Rubber Director. "Standard methods of compound evaluation and for reporting test data on GR-S tires", Rubber Age 52: 305-307 (1943)

Presence of Central Rubber Sheath around Cord

When tire cord is molded into rubber test-pieces for adhesion measurements the rubber invariably flows around the cord between the two rubber strips and usually completely envelops it. Rubber also flashes between the mold surfaces joining the two rubber strips, giving, in the final test-piece, a very thin connecting sheet of rubber. In conducting tests it has been customary to sever the connecting flash rubber next to that rubber strip from which the cord is to be separated in order to avoid any augmentation of the recorded test load due to this source. However, the rubber "sheath" which usually adheres tightly to the cord, is more difficult to separate before test without possible disturbance of the surface across which the adhesion is to be measured subsequently. This rubber envelope might introduce a very considerable discrepancy in the total measured force of adhesion. Experiments were, accordingly, undertaken to examine this question.

It may be supposed that any discrepancy in the adhesive force, caused by the rubber sheath, will bear some relation to the cross-sectional area of the rubber involved. Thus, since the cross section of the cord groove remains fixed and the cord cross section varies with the diameter of the cord, the discrepancy ought to be greater the smaller the cord diameter.

Three series of experiments were undertaken in which half the test pieces were tested in the usual manner and the other half were tested after carefully removing the rubber sheath. (See Figure 9.) The results, representing the mean differences in forces required before and after removal of the sheaths and adjusted for variations in cure, are summarized in Table 8. A few pieces were tested at both 75°F. and 275°F. without any cord having been present in the groove. These results, also, are recorded in Table 8.

Table 8. Excess Force Supported by Rubber Sheath During Adhesion Measurements.

Rubber stock	No cord in sheath		Cord Co-752 0.0286 in.		Cord Co-753 0.0319 in.		Cord Co-1356 0.0389 in.	
	At 75°F: Lbs.	At 275°F: Lbs.	At 75°F: Lbs.	At 275°F: Lbs.	At 75°F: Lbs.	At 275°F: Lbs.	At 75°F: Lbs.	At 275°F Lbs.
Co-1063:	2.43	0.32	3.95	1.79	2.89	1.59	-	-
Co-1063:	-	-	4.48	1.79	4.23	1.47	-	-
Co-1352:	-	-	3.15	1.11	2.30	1.34	2.56	1.23
Mean	2.43	0.32	3.86	1.56	3.14	1.47	2.56	1.23

By reference to Table 8, it will be seen that the rubber sheath causes a very considerable augmentation of the force required to separate the cord from the rubber. It was established by trials that these differences were not due to injury of the adhesion in the rubber strips where the

adhesion is intended to be measured. The sheath caused increases in the adhesion value at both room temperature and at 275°F. The amount of augmentation of the adhesion force in terms of the total was about the same at the two temperatures and in some cases exceeded 25 percent of the total. Also, as was predicted the sheath correction decreases as the gage of the cord increases, except for the case where no cord was present. Evidently, the presence of the cord influences the amount of augmentation. It is seen that the sheath effect is one of the most important encountered, thus far. Since it affects the absolute magnitude of the adhesion force, corrections will not be necessary in the evaluation of testing techniques, rubber stocks, etc., where the sheath effect may be considered to be the same in all samples to be tested. The method of treating this correction will be described in a later Section in connection with a discussion of "Characteristic Adhesion".

IV. INFLUENCE OF TIME AS A FACTOR IN THE TEST

Period of Time Elapsing between Curing and Testing

It has been the practice heretofore, among at least some of the laboratories using the "H" test for adhesion, to allow at least 24 hours to elapse after curing before the specimens are tested. Evidently it would be desirable, from the standpoint of expediency, to determine whether this aging period has a significant influence on the measured adhesion values, and is therefore necessary. It is likewise important to know whether the adhesion values of specimens not tested until several days after curing reflect this long delay. Often rigid testing schedules are difficult to maintain and such delays would facilitate testing if they did not influence the adhesion results.

In each of two experiments to compare the adhesion measured immediately after curing with that measured 24 hours later, two cures were prepared, one for testing at room temperature and the other at 275°F. The medium-gage cotton cord, Co-753, was used with GR-S rubber stock, Co-783. The curing pressure was 540 p.s.i., the other curing conditions being as indicated in the introductory remarks. Half of the specimens from each cure (alternate cords across the mold) were tested immediately. The remaining half of the specimens were stored at room temperature and tested 24 hours later. Also data were obtained from tests in which the times elapsing between curing and testing ran into several days. The same synthetic rubber stock and medium-gage cord and conditions of testing were used in these tests as in the specimens tested immediately and 24 hours after curing. The results are all presented in Table 9.

The results of Table 9 show surprisingly little influence on adhesion of delay in testing after cure. An analysis of variance showed that at room temperature adhesion is definitely influenced by elapsed time. A more detailed examination of the data showed that this effect was limited to the first 24 hours. In subsequent studies it was found that elapsed

time up to 34 days caused no change in adhesion values as compared with 1 and 7 days. Results obtained at 275°F. did not show any influence of delay after cure at any period.

Table 9. Force Required at Two Temperatures and at Different Intervals after Curing to Separate Cotton Tire Cord from Synthetic (GR-S) Rubber.

Interval after curing	Testing temperature	
	75°F.	275°F.
Hrs.	Lbs.	Lbs.
0	15.02 ± .20	6.40 ± .11
24	15.75 ± .18	6.37 ± .12
30	-	6.81 ± .25
52	15.35 ± .23	-
70	-	6.66 ± .17
72	15.63 ± .19	-
Average Force	15.44	6.56

The results given above are confirmed by other experiments employing natural as well as synthetic rubber. On the whole it may be concluded that the adhesion does increase for a time after cure, at least when measured at room temperature, but that it comes to a stable value during the first 24 hours after which it does not change appreciably. In a routine procedure it should be specified, therefore, that the actual measurement of the adhesion of specimens shall be delayed for at least 24 hours after curing. On the other hand, these experiments indicate that no upper limit need be placed on the period for testing. While the tests at 275°F. fail to show any effects of delayed testing on adhesion, it appears to be desirable, for uniformity and as a matter of precaution, to apply the same time-restrictions as are recommended for the room-temperature tests.

Length of Heating Period for Specimens at 275°F.

Early in this study of the "H" test a preliminary experiment was conducted to determine how quickly the interiors of the rubber end-strips attained a particular, selected oven temperature. Temperatures were measured by means of a chromel-constantan thermocouple inserted in the end-strips in the hole left by the cord, which had been pulled out. To simulate conditions under which the specimens would actually be heated, the strips under test were held in the regular clamps used for the adhesion test. The oven was preheated to the selected temperature before the rubber strip and clamp were placed in it. In Table 10 are recorded the results on a number of specimen end-strips heated at three different oven temperatures. The time-periods given are those required for the temperature (as given by the thermocouple) to attain an essentially stable value, after which the temperature fluctuations were of a minor character. A different end-strip was used for each trial at each oven temperature. The

results given in Table 10 suggest 1-1/2 hours as an adequate heating period to assure that the adhesion specimens attain uniformly a selected temperature in the range from 212° to 280°F. It is expected that a longer heating period would be required for final temperatures in a higher range.

Table 10. Heating Periods Required to Bring Temperature of End-strips of Adhesion Specimens up to Oven Temperature.

Oven temperature	Trial number			
	I	II	III	IV
°F.	Hrs.	Hrs.	Hrs.	Hrs.
221	1.00	1.08	1.33	-
248	1.03	1.05	1.03	-
275	1.00	1.33	1.50	1.33

The question arises as to whether after heating for 1-1/2 hours the actual adhesion attains a steady value associated with the preferred, high testing temperature and does not continue to decline as the heating period is lengthened. It would be desirable to know also whether the observed decrease in the adhesion in the specimens heated to 275°F. in part reflects an over-curing or degradative action which the rubber undergoes, permanently affecting its adhesive properties. In this case the adhesion measured after the specimens had cooled down to room temperature would differ from that measured on unheated specimens.

To investigate these points actual separation tests on adhesion specimens heated for 1, 1-1/2, 1-3/4, and 2 hours in an oven set at 275°F. were conducted. One set of specimens heated for each of these periods was tested immediately on removal from the oven, while corresponding sets were allowed to stand in the room for at least 24 hours after removal, before testing at 75°F. In molding the specimens, sample Co-753 cotton cord and a GR-S rubber stock were employed. The results are summarized in Table 11.

Table 11. Force Required to Separate Cotton Tire Cord from GR-S Rubber after Various Heating Periods in Oven Set at 275°F.

Heating period	Tested immediately on removal from oven	Tested 24 hours after removal from oven and at 75°F.
Hrs.	Lbs.	Lbs.
1	6.95 ± .16	14.95 ± .32
1-1/2	6.58 ± .14	14.97 ± .19
1-3/4	6.42 ± .17	14.52 ± .24
2	6.58 ± .17	14.74 ± .14
Control (not heated)	-	14.57 ± .40

The adhesive forces of specimens tested immediately on removal from the oven show a slight drop between 1- and 1-1/2-hour heating periods. This difference is likely to be real, indicating that after only 1-hour heating the specimens have not quite reached the prevailing oven temperature. The variations in the adhesive forces of specimens heated for 1-1/2 hours and longer are not significant, though the differences between these results and those for 1-hour heating are significant. The adhesion of specimens allowed to return to room temperature before testing shows no effect resulting from the heating, even in the specimens heated for two hours. There are no statistically-significant differences among the five mean forces measured at 75°F. These results further justify the decision that 1-1/2 hours is the optimum heating period for the test at 275°F. Since heat during this period evidently does not permanently damage the specimens, the test measures only the physical effect of high temperature on adhesion of cotton to GR-S rubber.

Delay in Testing Hot Specimens

The time elapsing between the removal of a specimen and clamp from the oven and the actual separation on the machine could conceivably affect the adhesion measurement in the high-temperature tests. This operation ordinarily requires 15 to 20 sec. per specimen, but if any difficulty is encountered in positioning the specimen, or tightening the upper clamp, or if the operator is inexperienced, the time required may easily be doubled. In this interval the specimen is obviously cooling off. The question is how quickly the actual separation must be made if the results are to be sensibly reproducible.

An experiment to study this point was conducted, using natural rubber stock, Co-767, and cotton cord, Co-753. The specimens were heated for 1-1/2 hours in an oven at 275°F., and were removed one by one for the breaking test. Each specimen and clamp was allowed to lie on the table at room temperature for the desired delay period, which was measured from the instant that the specimen was removed from the oven. Eleven or twelve observations at each delay period were obtained. The results are given in Table 12.

Table 12. Force Required to Separate Cotton Tire Cord from Natural Rubber in Specimens Tested at Various Times after Removal from Oven.

Delay period	Force
Min.	Lbs.
0	7.12 ± .18
1	7.35 ± .12
5	8.80 ± .20
90	13.69 ± .30
Control (not heated)	15.04 ± .56

The analysis of variance in the data indicates that a delay in testing up to about one minute introduces no significant difference (error) into the high-temperature adhesion tests. For delay periods longer than this the observed adhesion forces evidently rise, until after 90 min. the specimens regain about 85 percent of the loss in adhesion at high temperature. Results in the preceding section (though not on natural rubber) suggest that the 15 percent remainder is not a permanent loss, but requires more than 90 min. for recovery.

V. RUBBER-STOCK FACTORS INFLUENCING ADHESION

Differences between Rubber Stocks

Reference has been made in the section on "Testing Temperature" to the differences in adhesive behavior between natural and GR-S rubber stocks, shown in Figure 3. Differences between the three samples of GR-S rubber are also evident in the figure, though they are minimized at 75° and 275°F. It is to be expected that the adhesion of tire cord will be different also for rubber stocks of the same general type but from different sources, or from batches compounded at different times. Even where duplication is attempted, random differences in composition will enter, and these may be sufficiently pronounced to be reflected in significant alteration of the adhesion in one direction or the other.

The two GR-S rubber stocks, Co-768 and Co-783, for which results are given in the foregoing Table 7, were from two different sources, and hence, close uniformity in composition was not to be expected. A detailed statistical examination of the apparent differences in adhesive values between these two stocks cured in the Armstrong mold, revealed that these differences are probably statistically significant when the two stocks are used in conjunction with the medium-gage cord and tested at 75°F. When the low-gage cord was employed and the adhesion was measured at room temperature, or when the adhesion of either gage of cord was measured at 275°F., the two stocks did not give significant differences. In this connection it should be recalled that the curing conditions for these two stocks were not identical, though they are believed to have been equivalent.

Time and Temperature of Storage of GR-S Stock

When the experiment on rubber stock Co-1063 showing the influence of testing temperature on adhesion, as set forth in Figure 3, was repeated 11 weeks later, it was found that the force to separate was slightly higher at all eight testing temperatures. Repetitive tests on stock Co-783 at 75° and 275°F. (under conditions identical within experimental control) also suggested an improvement in the adhesive properties of GR-S stock on standing. In all these cases the stock was stored in an electrical refrigerator at an average temperature of 45°F. The question naturally arose, therefore, as to whether the apparent increase was real,

i.e., whether stock kept at refrigerator temperatures for some weeks gives significantly different results in adhesion tests after such storage. The related question was whether cold storage was necessary to offset or minimize changes in the adhesive behavior which might be found to occur in GR-S stock stored at room temperature.

To investigate these points, samples were taken from the outer end of the roll of rubber stock, Co-1352, immediately after receipt. Cotton tire cord Co-753 was used throughout. One sample of the rubber was cured to the cord immediately. The other samples were divided into two equal parts and one half was stored at room temperature (about 75°F.), the other at 45°F. Portions of the samples from each storage temperature were removed at the end of 33 and 61 days and cured to the cord. The results are presented in Table 13.

Table 13. Force Required to Separate Cotton Tire Cord from GR-S Rubber Stock Stored at Room Temperature and at 45°F. for Different Periods.

Time of storage	Temperature of storage	Force at 75°F.	Force at 275°F.
Days	°F.	Lbs.	Lbs.
1	45	18.75 ± .35	7.40 ± .10
33	75	17.32 ± .36	6.87 ± .07
33	45	17.94 ± .33	7.13 ± .20
61	75	16.40 ± .18	7.21 ± .08
61	45	18.15 ± .22	7.71 ± .14

By reference to Table 13 it will be seen that the rubber stock kept at 45°F. in the refrigerator shows neither a gain nor an appreciable loss in adhesive property, whether measured at room or at elevated temperature. Statistical analysis of the data, revealing that the differences are not significant, confirms this conclusion. On the other hand, it will be seen that the adhesive force measured at room temperature declines steadily if the GR-S stock is stored for long periods at room temperature before curing; the adhesive force measured at 275°F. appears not to be affected. Analysis of the variance in these data shows that the storage temperature is highly significant. It is evident, therefore, that storage of the uncured GR-S stock at a low temperature is necessary if reproducible results are to be obtained.

Uniformity of Adhesion within a Single Roll of GR-S Stock

In conjunction with the tests described in the preceding section, measurements were also made on specimens molded from GR-S rubber, Co-1352, taken from the middle and inner end of the same roll. These specimens were molded from rubber samples which had been stored at room temperature, or in the refrigerator at about 45°F., for 33 days after receipt of the roll. Cord Co-753 was used in building the specimens; curing and testing conformed to the usual procedures. The results of this experiment are summarized in Table 14.

Table 14. Force Required to Separate Cotton Tire Cord from GR-S Rubber Taken from Different Parts of Roll of Stock and Stored for 33 Days.

Original position in roll	Temperature of storage	Force at 75°F.	Force at 275°F.
	°F.	Lbs.	Lbs.
Outer end	75	17.32 ± .36	6.87 ± .07
Middle	75	16.37 ± .33	6.69 ± .17
Inner end	75	16.15 ± .30	6.30 ± .33
Outer end	45	17.94 ± .33	7.13 ± .20
Middle	45	17.04 ± .36	7.00 ± .21
Inner end	45	17.57 ± .35	7.25 ± .16

On the first perusal of the data set forth in Table 14 no consistent differences in adhesive behavior between GR-S samples from different parts of the roll are discernible. It is seen, furthermore, that the differences in most cases are small among samples stored at the same temperatures and tested at the same temperatures. Analysis of variance of the data in Table 14 shows that the difference in adhesive properties associated with different positions in the roll has some slight significance. However, none of the differences between mean forces in particular tests made on rubber stored at 45°F. is found to be statistically significant. It may be concluded then that the sample of GR-S rubber stock, Co-1352, used in the present experiment, was substantially uniform throughout the length of the roll, insofar as its adhesive properties are concerned.

VI. CORD FACTORS INFLUENCING ADHESION

Gage of Cord

In the determination of adhesion of tire cord to rubber carcass stocks, cords of differing gage invariably will be encountered. That gage influences the adhesion value was shown clearly by an analysis of variance in the original data of Table 3. Hence, account must be taken of the gage in assessing the merits of different factors that influence the adhesion, when cords of different gage are employed.

The adhesion may be supposed to be proportional to the relative surfaces involved. Thus, we may expect

$$A = k\pi GL \qquad (1)$$

where A is the measured force of adhesion, G is the measured gage of the cord, here used as diameter, L is the contact length and k, the proportionality factor, is the adhesive force per unit area. It was accordingly

of interest to determine whether this relationship held approximately. We shall not anticipate too close an agreement since products such as tire cord are quite variable in diameter and actual twist, even within short intervals. Furthermore, in the cords here employed there were unavoidable differences in twist as well.

According to the preceding formula since the length of contact of cord in a test piece is constant at 3/8 inch, it will be evident that the adhesion should be proportional to the apparent circumference, i.e., to the gage of the cord multiplied by π. Hence the ratios of adhesive force to gage should be constant for a given cotton and construction of cord. The gages of each length of cord mentioned in the preceding Section on "Pressure on Mold during Cure" having been measured, the ratios of adhesive force to gage were computed after combining the original data summarized in Table 3 for the two higher pressures. This combination was legitimate since it was found that pressures of this order had no influence on the adhesion within the precision of measurement. The ratios thus computed are recorded in Table 15 according to temperature of measurement and mean gage of cord employed.

Table 15. Ratio of Force of Adhesion to Gage for Two Tire Cords of Different Gages in GR-S Rubber Tested at Two Temperatures.

Tested at 75°F.		Tested at 275°F.	
Low gage (0.0290)	Medium gage (0.0312)	Low gage (0.0290)	Medium gage (0.0312)
Lbs./In.	Lbs./In.	Lbs./In.	Lbs./In.
417	472	208	211
448	471	238	207
490	478	203	206
510	502	237	239
436	485	252	251
458	495	251	244
483	488	205	220
507	535	193	172
485	522	215	203
456	472	214	220
448	533	248	245
427	428	221	231
466	503	216	213
449	488	219	188
		176	168
		208	189
462.9	490.9	219.0	212.9
Mean 476.9		216.0	

By reference to Table 15 it will be seen that, for the two gages of cord used, the mean ratios based on adhesion measurements at room temperature differ considerably from each other while those based on measurements at 275°F. show rather better agreement. It will be observed that very considerable variation in the individual ratios is found. These are doubtless caused by the correspondingly large variations in cord gage, even within the short lengths employed, as well as other uncontrolled variations referred to above. The range of gage of the low-gage cord was 0.0282 to 0.0299 inch and of the medium-gage cord 0.0301 to 0.0324 inch. The lengths of cord used were about 8 inches and the length of contact between rubber and cord only 3/8 inch. It was not practicable to measure the gage of the cord preceding cure at the exact position where adhesive contact was to be made with the rubber.

In order to assess the significance of the above mean differences an analysis of variance was made. If the differences associated with gage are not significant we may assume that Equation (1) satisfactorily represents the existing relationship, within the precision of the data. The results showed that the ratios obtained by dividing the adhesion by the gage do not differ significantly with gage even though the adhesive force values themselves do differ significantly. We may accordingly assume satisfactory verification of the formula.

In a second experiment to obtain a more precise evaluation of the influence of gage on adhesion, a special high-gage cord, Co-1356, was prepared with a construction of 17/5/3 and giving a mean gage of 0.0389 inch. The cord twist used (8.84 t.p.i.) was very nearly the same as that of Sample Co-753 (8.7 t.p.i.) which was also used, along with Sample Co-752. The rather large range of gage used in this experiment was thus from 0.0286 to 0.0389 inch. The cords were vulcanized in rubber stock Co-1352 and the adhesion measured at room and at elevated temperatures. The means of the results are shown in Table 16.

Table 16. Force Required to Separate Tire Cords of Widely Different Gage from Synthetic Rubber Stock (Co-1352) at Two Temperatures.

Cord sample	Mean gage	Tested at 75°F.	Tested at 275°F.
	Inch	Lbs.	Lbs.
Co-752	0.0286	16.26 ± .28	5.92 ± .18
Co-753	0.0319	18.06 ± .26	7.33 ± .13
Co-1356	0.0389	23.55 ± .22	9.34 ± .18
Mean	0.0331	19.29	7.53

By reference to Table 16 it will be observed that increase of gage results in a progressive increase in the force required to effect separation. In Figure 5 the averages of adhesive force at the two temperatures have been plotted against gage, and curves have been drawn in,

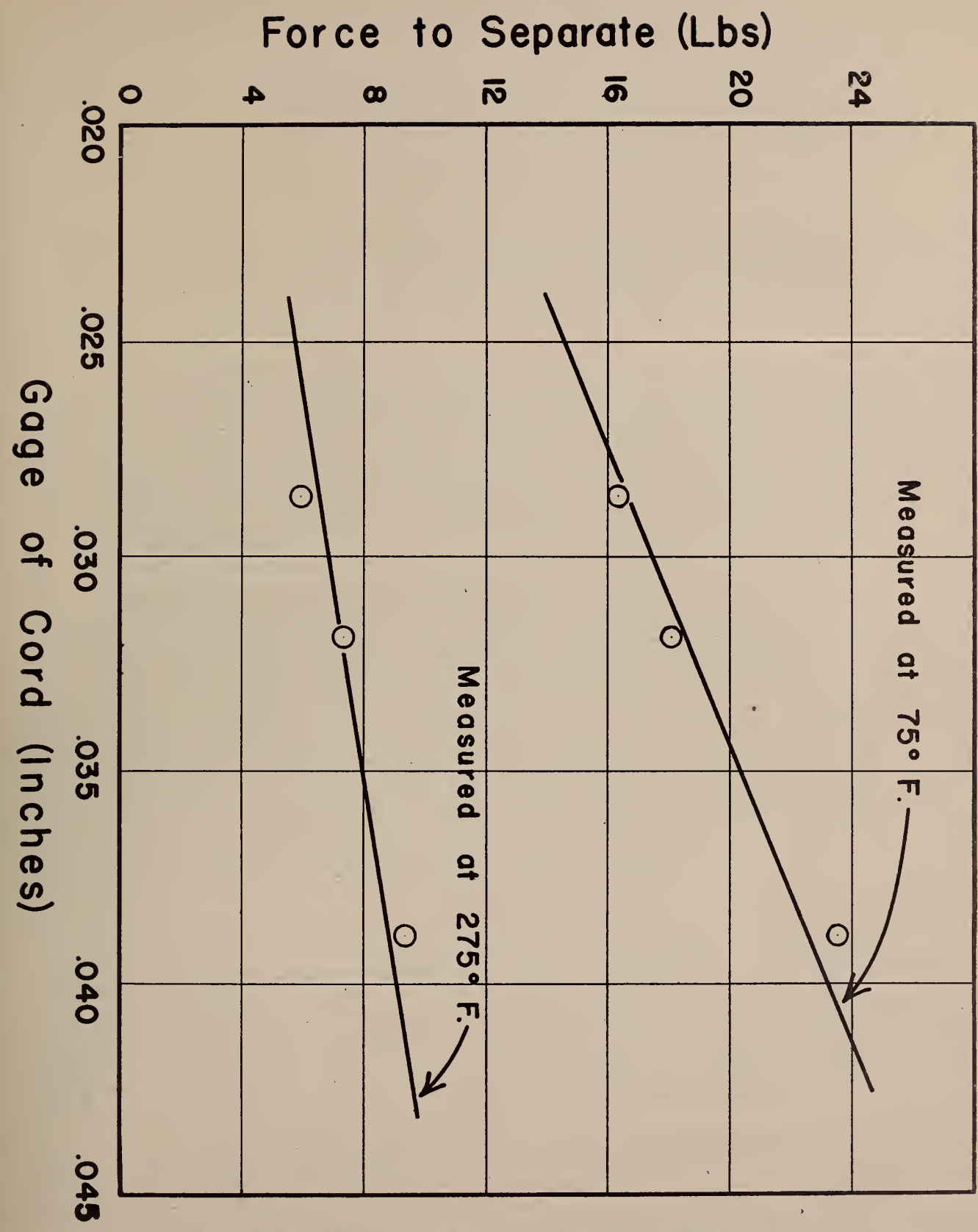

Figure 5. Effect of Gage on Rubber-Cord Adhesion.

corresponding to the theoretical relationship, which assumes a direct proportionality between adhesive force and cord surface. The data show reasonable agreement between observation and theory, there being a slight tendency for adhesion to increase more rapidly than gage.

As a result of these experiments it is evident that whenever the adhesion of cords of different gage is to be compared adjustment must be made for the difference in gage. The method of making this adjustment will be discussed in Sec. VII, dealing with "Characteristic Adhesion".

Number of Plies and Cord Twist

It may be supposed that the degree of twist in the cord will influence the adhesion. Furthermore, the opinion has sometimes been expressed that a 2-ply cord will have greater adhesive strength than a 3-ply cord of the same twist. In considering twist it is quite customary in the tire industry to refer to the turns per inch of the plies which give rise to the final cord. It will be obvious, however, that for the same number of turns per inch a 2-ply cord will have a lower cord angle (helix angle) than a 3-ply cord.

In order to obtain cord samples in an adequate range of twists, lengths of the medium-gage cord Co-753 were given increased and reduced twist so that this extended greatly above and below that in customary use in tire cord. Also, portions of this same cord were untwisted with the aid of a twist counter, one ply was removed and twist was then reinserted, so as to give about the same number of turns per inch as for the 3-ply cord. Also portions of the low-gage cord, Co-752, were given reduced or increased twist comparable to that inserted in the 3- and 2-ply cords described above. The cord angle of the cord samples was measured with the aid of a Leitz petrographic microscope at 20 diameters, and was found to vary from 17.5° to 46.3°. The cords were cured into synthetic rubber stock, special care being taken not to lose the added twist, and the adhesion was determined at room temperature and at 275°F.

The results were presented in Table 17 as "characteristic adhesion" a quantity which will be discussed in more detail below. The results are also given as percentages of the adhesion of the 23/5/3 control cord, Co-753. By reference to the table it will be seen that the twist varied from approximately 7 to 16 turns per inch -- well beyond the conventional practice. It will be observed from the columns of percentages that at both room temperature and 275°F., there is a slight tendency for adhesion to increase with twist, but the effect is surprisingly small.

In general, the 2-ply cord does not show better adhesion than the 3-ply cord. This is brought out more clearly in Figure 6, where the characteristic adhesions in Table 17 are plotted against the cord angle. It is apparent that the distribution of the points about the best-fitting straight lines permits no distinction to be drawn between 2- and 3-ply cords with reference to adhesion. It may be concluded that within the ranges of gage and twist (cord angle) embraced by the present study the adhesion of cotton cord is independent of the number of plies. The trends in Figure 6 confirm the observation that adhesion increases slightly with twist.

Table 17. Influence of Number of Plies, Twist, and Gage of Cord on Adhesion.

Sample number	Plies[1]	Twist[2]	Cord angle	Cord gage	Characteristic Adhesion K			
					At 75°F.		At 275°F.	
					Absolute	Relative[3]	Absolute	Relative[3]
	No.	t.p.i.	Degrees	Inches	p.s.i.	Percent	p.s.i.	Percent
Co-1334	3	7.17	22.6	0.0306	1159	101	458	92
Co-753	3	9.03	28.7	.0315	1147	100	498	100
Co-1335	3	10.90	34.8	.0300	1167	102	498	100
Co-1336	3	12.73	38.5	.0303	1203	105	527	106
Co-1337	3	16.37	46.3	.0297	1214	106	514	103
Co-1338	2	7.25	17.5	.0264	1047	91	414	83
Co-1339	2	9.25	21.0	.0258	1090	95	432	87
Co-1340	2	11.10	23.7	.0250	1157	101	463	93
Co-1341	2	13.05	29.6	.0245	1107	97	445	89
Co-1342	2	15.70	35.3	.0245	1185	103	467	94
Co-1343	3	6.87	18.5	.0273	1032	90	445	89
Co-752	3	10.58	33.1	.0283	1188	104	462	93
Co-1344	3	13.10	35.4	.0266	1234	108	466	94
Co-1345	3	15.80	42.7	.0271	1204	105	498	100

1/ The 2-ply cords were made from Sample Co-753 by untwisting, removing one ply and retwisting to the desired number of turns.

2/ Measured with a thread-counter after retwisting.

3/ Based on value for control cord Co-753 taken as 100 percent.

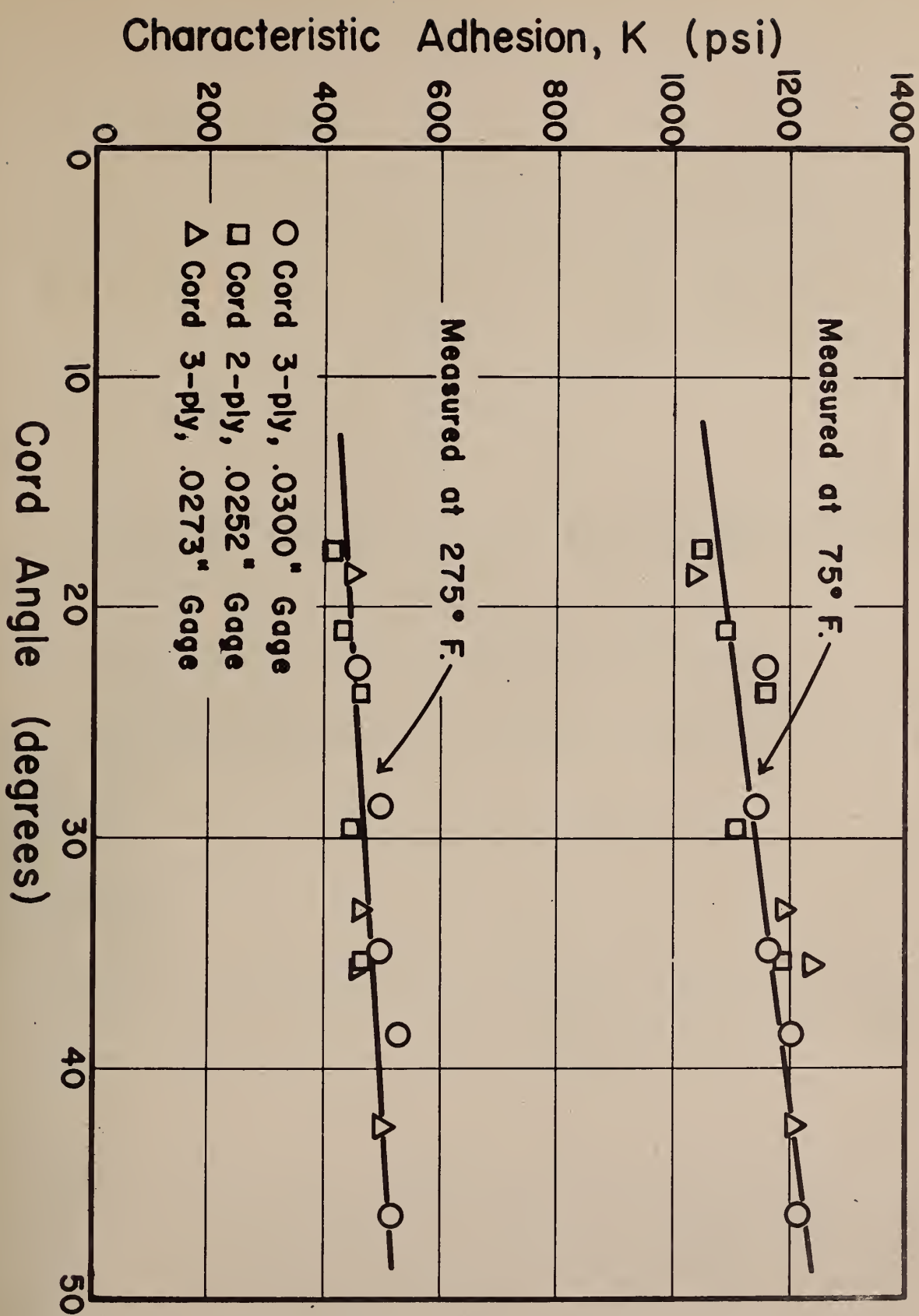

Figure 6. Characteristic Adhesion as a Function of Cord Angle at Two Testing Temperatures.

Adjustment for Cord Twist

A relation by means of which the adhesion of a cord having a given twist could be calculated from observations on a similar cord having some other twist would be useful in many cases. While the evidence to which reference has just been made favors the cord angle rather than the turns per inch for adjustment of different constructions, the former is less convenient in practice. Since the effect is quite small, adjustments may be made in characteristic adhesion with sufficient accuracy by means of a relation involving the turns per inch. Applying the method of least squares to the data in Table 17 for the measurements at room temperature, the following equation has been derived:

$$K_2 = K_1 + 13.7\ (T_2 - T_1), \qquad (2)$$

where K_1 and K_2 are the available and desired characteristic adhesions, respectively, and T_1 and T_2 are the corresponding turns per inch in the cord. For the characteristic adhesion measured at 275°F. the corresponding equation is

$$K_2 = K_1 + 5.6\ (T_2 - T_1) \qquad (3)$$

where the symbols have the same significance as before.

Equations (2) and (3) show the small increase in adhesion with twist; at room temperature the average increase per turn-per-inch is only 13.7 p.s.i., while at 275°F. it is 5.6 p.s.i. per turn-per-inch.

VII. THE CHARACTERISTIC ADHESION

Definition

In Sec. VI it was pointed out that the adhesion varied with the gage of the cord being tested and this was traced to variation of the area of adhesive surface with gage. It was shown that when the measured adhesion was expressed as a ratio of the cord surface involved, the significant effect of gage disappeared. Thus, from Equation (1) we have

$$\frac{A}{G} = k\pi L \qquad (4)$$

where the left-hand term characterizes the adhesion, in shear, of a particular cord, independently of the gage. Ideally, it measures the force required to separate from a rubber strip at one time, all the cords lying side by side as closely as possible in a 1-inch strip of fabric, and imbedded in the rubber for a distance L along the cord axes. Since evidently the ratio A/G is proportional to L we may refer the ratio to unit length of contact (1 inch) by dividing by L:

$$\frac{A}{GL} = k\pi = K \qquad (5)$$

Insofar as the observed value, A, gives the force actually required only to shear the cord-rubber bond of one cord along the length L, the quantity $k\pi = K$ measures the adhesion of a particular cord-rubber combination independently of gage and length of contact. It may be interpreted physically, in the ideal case, as the force required to shear a 1-inch width of tightly packed cord fabric from a 1-inch square of rubber. Since the length of contact of cord to rubber, after removal of the rubber sheath around the cord between the end-strips, is always 3/8 inch in the present "H" test, we have

$$K = \frac{8A}{3G} \qquad (6)$$

where A and G are the measured adhesion (in pounds) and cord gage (in inches), respectively. K may be called the "characteristic adhesion".

As an example of how "characteristic adhesion" can be applied to tire carcasses containing a specified number of cords per inch, the characteristic adhesion, as determined above, may be multiplied by the ratio of the specified to the greatest possible number of cords per inch, to give a practical estimate of the adhesion of a particular fabric. Thus, with a cord having a gage of 0.030 inch, and a K value of 1150 p.s.i., to be used in a tire with 24 ends per inch in the ply, this fabric adhesion K_f may be calculated:

$$K_f = 1150 \times \frac{24}{1/.030} = 828 \text{ p.s.i.}$$

If instead of 24 ends per inch 28 ends were used, the fabric adhesion would be increased to 966 p.s.i.

Estimation of Characteristic Adhesion from Tests on Specimens with Sheath Present

In the foregoing discussion of characteristic adhesion, it was assumed that the rubber sheath around the cord between the end-strips had been removed before the specimens were tested. In some cases, however, the procedure followed may not involve removal of the sheath. Considerable data were collected in the present study before the magnitude of the effect of this sheath on the measured adhesion was fully appreciated.

Evidently, if comparisons between results on the two types of specimens are to be made, a relationship is required by means of which the gross or crude characteristic adhesion, computed for the specimens with the sheath present, can be converted to the true characteristic adhesion, such as would have been derived had the sheath been removed from the specimens. The data already on hand were amplified by determination of the characteristic adhesion with and without removal of the rubber sheath from both cotton and rayon tire cords. Both untreated cords and cords treated with adhesive compounds were included in the test. The usual procedures for curing, preparing and testing the specimens were followed. GR-S rubber stock was used throughout the series of tests, and adhesion

was measured at both room temperature and at 275°F. At room temperature 32 pairs of means each of 10 or more observations, and at 275°F. 34 pairs of means were employed. The gage of the cords varied from 0.022 inch to over 0.038 inch. Since it had been found that gage influences the sheath correction, gage was made one of the independent variables also in this correlation analysis. The results are set forth graphically in Figures 7 and 8, for the measurements made at room temperature and at 275°F., respectively.

By reference to Figures 7 and 8 it will be seen that while considerable scatter is present, in general, the corrected characteristic adhesion is positively related to the crude characteristic adhesion, K_u. In line with expectation, based on Table 8, the gage of the cord plays a small but definite part. The regression equations computed by the method of least squares, assuming a linear relationship, are, for characteristic adhesion at 75°F.

$$K = 0.8960\ K_u + 5395\ G - 299, \qquad (7)$$

and for that at 275°F.

$$K = 0.5518\ K_u + 3305\ G + 65, \qquad (8)$$

the gage G being expressed in inches. Further analysis of the data showed that the correlation coefficient associated with Equation (7) was 0.934 and the standard error of estimate 70.3 p.s.i. For Equation (8) the corresponding figures were 0.760 and 58.6 p.s.i., respectively. Regression lines corresponding to the gages 0.024 inch and 0.032 inch are drawn in, in Figures 7 and 8.

Some question arose as to the correct gage measurements to be used for G in Equations (5), (6), (7), and (8) as applied to adhesive-treated cord samples. One approach to this problem was to recalculate the characteristic adhesions K and crude adhesions K_u given in Figures 7 and 8 using for the adhesive-treated cords, the gage of the untreated control. When K and K_u were correlated on this basis, the correlation coefficient for the tests at 75°F. was raised very slightly to 0.972, while that for the tests at 275°F. was lowered very slightly to 0.752. While the small over-all improvement here might seem to dictate the use of the gage of the untreated control cord for G, in the relevant equations, a consideration of the physical significance of K and K_u discloses that the use of such a gage measurement would lead to erroneous conclusions concerning the practical value of a particular adhesive.

As pointed out in the discussion following Equation (5), K measures the adhesive force of a 1-inch strip of tightly packed cord fabric. In setting up Equation (5), it was supposed that the 1-inch width of fabric contains the maximum number of cords possible, viz., 1/G per inch width. Evidently G here must include any extra width the cord may have acquired as a result of the addition of adhesive. The use of a smaller G, as that of the untreated cord, in effect assumes that there are more cords per inch than can actually be laid in one inch, and accordingly, leads to a

fictitiously high value for K. It is clear, therefore, that the measurement for G for adhesive-treated cords must be made on the actual treated cord sample. This procedure has the further advantages that the use of Equations (7) and (8) is possible on adhesive-treated samples for which no untreated control is available.

In summary, it should be noted that by definition, the term characteristic adhesion applies to results on specimens from which the rubber sheath has been removed. Formulas (7) and (8) are for conversion of original observations made on specimens from which the sheath was not removed.

VIII. PROPOSED PROCEDURE FOR CONDUCTING THE "H" ADHESION TEST

Rubber Stock and Preparation of Molded Test Specimens

The rubber skim stock, both natural and synthetic, cut into sections approximately 10 x 12 inches, is stored at low temperature, preferably in a refrigerator. Before use, each new supply is to be gaged with a dial micrometer under 4-ounce pressure to make sure that when properly cut it will completely fill the mold channels and allow for some overflow during cure. The Holland cloth is not included in this gage. Skim stock for use in the Armstrong mold, having the backing fabric already "frictioned" and calendered onto the rubber, should have a gage of about 0.055 inch, and should be rejected if the gage falls below 0.050 inch since the channels themselves are 0.1 inch deep and two layers would allow for no overflow. Skim stock for use in the Mansfield mold, having an unfrictioned 8.6-ounce duck backing cloth loosely laid on the rubber, should have a gage of about 0.095 inch including canvas to allow for overflow since the backing cloth itself must be impregnated and the channels in this mold are 0.125 inch deep. The skim stock sections should be gaged from time to time as they are used, to insure completely full channels during cure.

The mold with cover, previous to curing, is placed between the hot plates of the hydraulic press and allowed to heat up with the plates as steam passes through the latter.

In the meantime, with a double-bladed tool in which the blades are accurately spaced to match the mold channel width[13], strips of skim stock are cut 10 inches long for the Armstrong mold (or 8 inches long for the Mansfield mold). These are cut from the side having the backing fabric, and usually with the length parallel to the warp of the fabric, though the directions of warp and filler in the rubber strips were not found to be important. Twelve such strips are required for each cure in either the Armstrong or Mansfield mold.

13/ It appears allowable to increase the width of skim-stock strips slightly when doing so will compensate for a slight deficiency in the thickness of the stock which would otherwise result in no overflow and a loose cast.

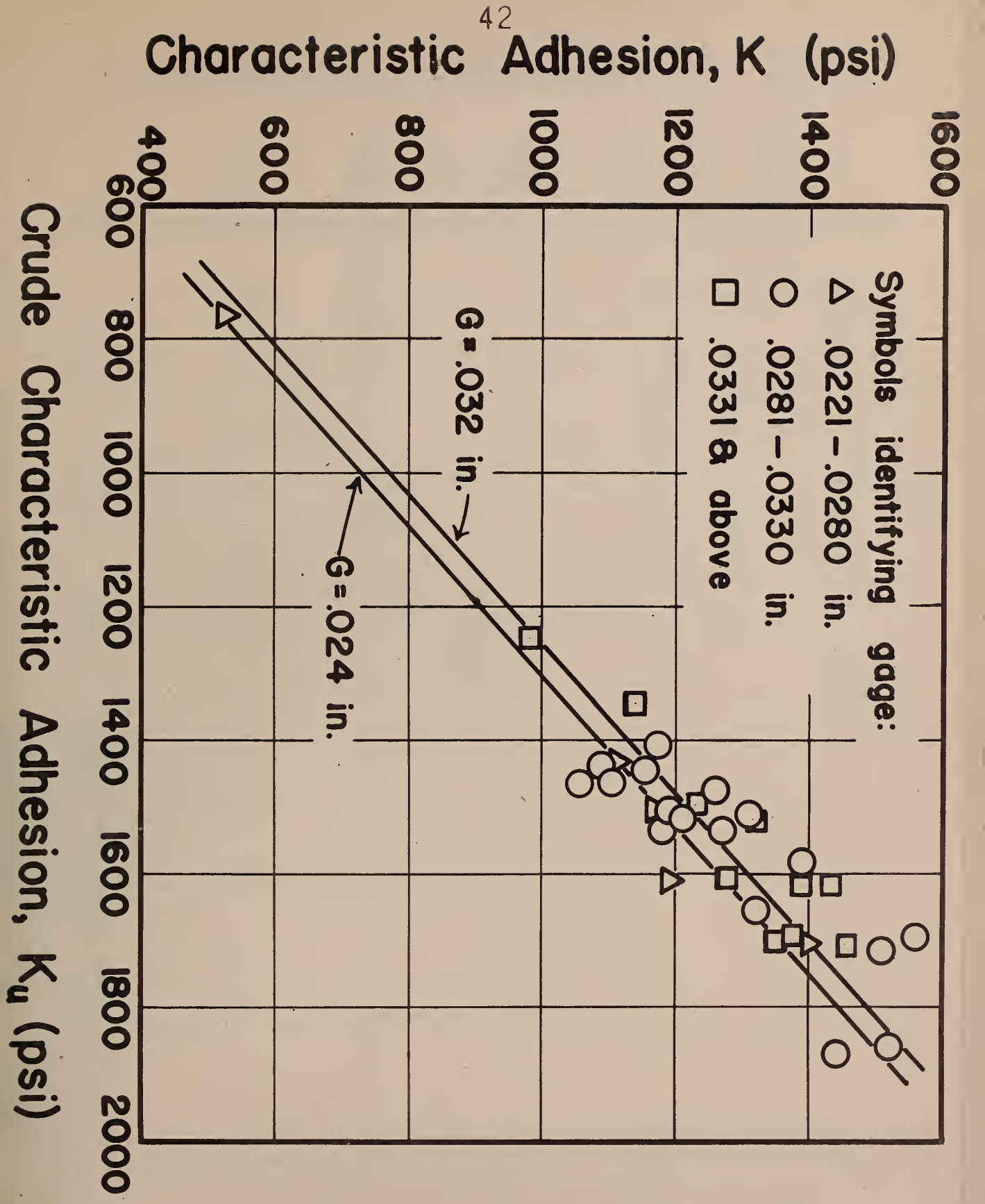

Figure 7. Characteristic Adhesion, K, as a Function of the Crude Characteristic Adhesion, K_u, (Sheath Present), Determined at 75°F.

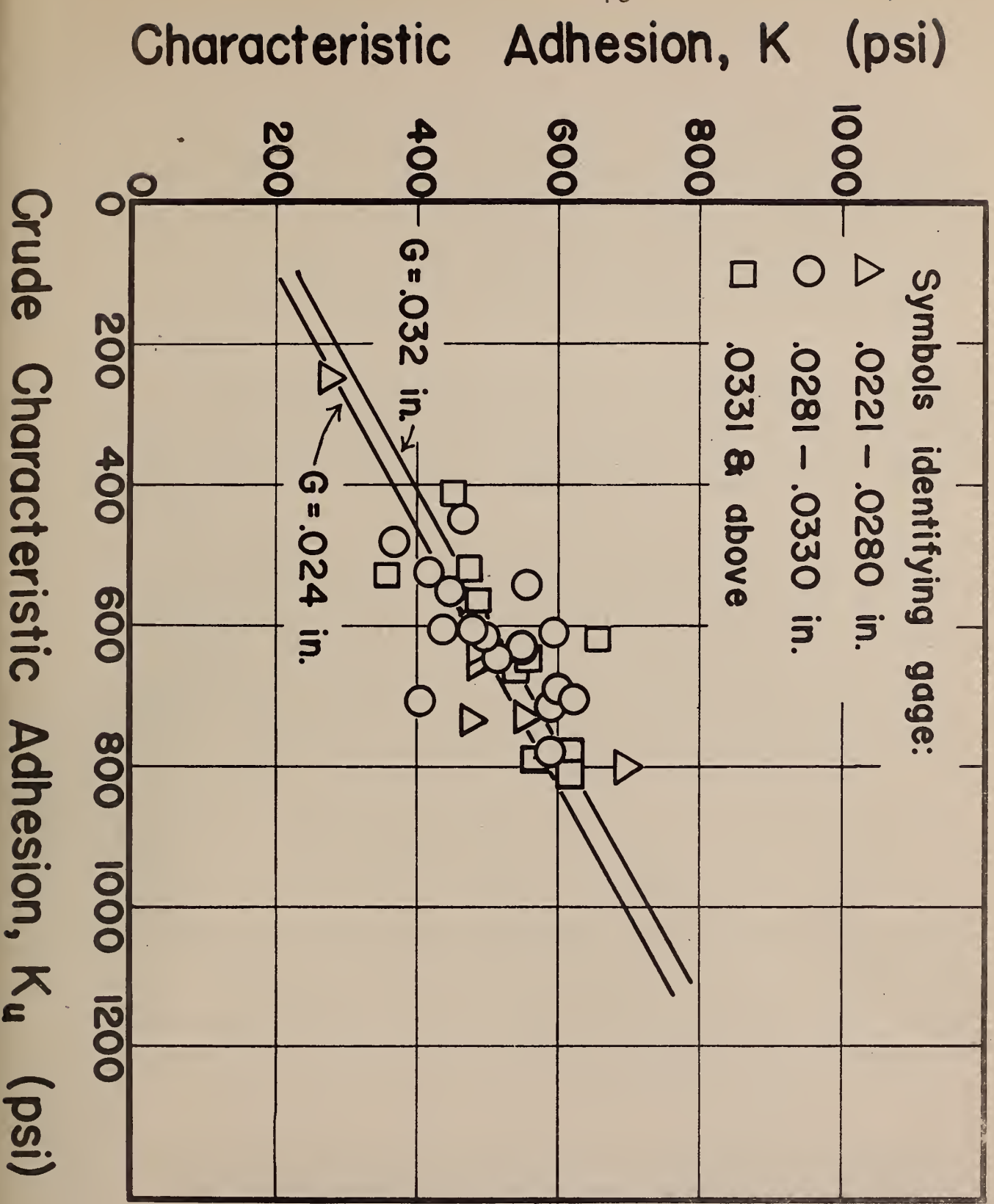

Figure 8. Characteristic Adhesion, K, as a Function of the Crude Characteristic Adhesion, K_u, (Sheath Present), Determined at 275°F.

Some time before the molding operation is begun, or while the mold is being preheated, the gages of the cord samples to be tested are obtained according to conventional methods, and recorded. The cords are to be in a normal condition of humidity, i.e., not exposed to relative humidities above 60-70%. Cord specimens should be about 10 inches long for the Armstrong mold and 16 to 18 inches long for the Mansfield mold. Before being cut to these lengths the cords should be knotted so that when the cuts are made a knot will fall about a half inch from each end of each specimen (this procedure is followed to avoid loss of twist in the cord during handling).

In addition to the regular test cords it is advantageous to have in every test a "basic control" cord. This cord should be chosen with respect to high uniformity, and similarity with the cords that are to be studied, insofar as possible. The use of this control permits a very considerable improvement in the comparison of adhesion results obtained over a period of time.

When the press and mold have come up to the required temperature of 287°F. and have remained there for about 15 minutes, the mold is removed to a table. The rubber strips are pulled one by one from the Holland cloth and quickly fitted, with fabric side down, into the six channels in the mold. The specimen cords, properly identified[14/] are now laid in the eight or ten transverse cord slots in the mold, so as to cross the skim coat of the rubber strips. Each cord is held in place by the end knot resting against the back edge of the mold. A four-ounce weight is clipped to the other end of the cord hanging down over the edge of the mold and table, in order to provide uniform tension on the cord.

Special care should be taken in routine tests to distribute cord lengths from each test sample and control among as many cures as is feasible, since it is possible that the cure itself gives rise to some variability in results; i.e., the mold in any single cure is not to be filled with cord from a single sample and in the next cure with a second; but both cures should contain both cords. To provide a number of test pieces adequate for adhesion tests at room as well as at elevated temperature, as many cures are required as there are samples and controls. In general, more than this number of cures will not be made. Cords from different samples should be located progressively in the successive grooves of the mold in successive cures so that in the whole series of cures each sample will have occupied as many of the different grooves as possible.

14/ In cases where numerals are engraved in the mold so as to identify each test piece as to slot and channel, record of the particular cord sample in each test piece may be kept by means of a table in which is entered under each slot number the sample number of the cord in that slot in the particular cure.

When all the cords are in place, the remaining six strips of rubber are pulled from the Holland cloth and laid with the skim coat down in the channels directly over the strips already in the mold. The last six strips are pressed into place so as not to overlie the edges of the channels at any place. The cover is replaced on the mold, the weights removed from the cords, the protruding ends of the cords are clipped off near the edge of mold; and the assembly is placed in the press, all as quickly as possible. The rubber is cured for 30 minutes at 287°F. (or at the proper temperature for the stock) under a load computed to give a pressure of 250 p.s.i. on the surface of the mold.

When the curing has been completed, the grid-like rubber molding or cast produced is set aside for at least 24 hours before the actual breaking tests are started.

Separation of Specimens and Measurement of Adhesion

The rubber casts produced in the cures are cut into specimen preparatory to testing, each providing 24 or 30 of the H-shaped test specimens, the number depending on whether the Mansfield or Armstrong mold is used. Half of the specimens from a particular series of cures are used for adhesion tests at room temperature, while the other half are set aside for tests at an elevated temperature.

Before actual testing (or before heating in the 275-degree tests), the rubber sheath around the cord, and the web of "flash" extending between the rubber end-strips of each test specimen, are removed so as not to contribute a spurious increment to the actual adhesive force. This operation is shown in Figure 9. The specimen is grasped with that end-strip that will go into the upper jaw between the thumb and index finger of the left hand, and the end-strip from which the cord will be pulled, merely hanging free. With a sharp forceps the rubber is first broken from about the cord at a point near the thumb, and when broken loose is stripped continuously toward the other freely-hanging end-strip. When the rubber sheath has been separated just down to this free end-strip, it is released and left hanging loose. Great care should be observed in this operation that no stresses are put on the cord-rubber interface in the end-strip from which the cord will be pulled subsequently. The rubber about the cord at the point where the latter enters the end-strip should not be disturbed.

All adhesion tests are to be made on a vertical pendulum-type, rubber or textile testing machine. The speed of the lower moving clamp is to be fixed at 12 inches per minute. Because most of the significant adhesion separations will occur in the range of force from 5 to 20 pounds, a testing machine having a capacity in excess of 50 pounds should ordinarily not be used. If spark or ink recording on a paper chart is used care is taken to see that the dial and chart readings agree. Also, with the pointer on the dial reading zero the zero position on the chart should be recorded with ink or the spark for purposes of reference.

Figure 9. Technique for Removing Rubber Sheath and Web of "Flash" Between Rubber End-strips.

Both the upper and lower clamps are one inch wide (the same as the length of the end-strips of the test pieces). The upper clamp, permanently attached to the upper tension bar or chain of the machine during a series of tests, has a continuous clamping surface across the whole width of each jaw. The lower clamp, as shown in Figure 10, fits on a stud on an adapter through which the breaking force is applied. This clamp has a vertical slot about 3/16 inch wide in the middle of both jaws. In fitting the clamps to the machine they are adjusted so that the distance between the horizontal edges of the jaws in the starting position is 1/4 inch, the distance between the rubber strips in the test pieces.

In the tests at room temperature, only one lower clamp is used throughout a series of tests. The prepared test pieces of a particular sample are inserted in the clamps and tested consecutively. Care is taken that the cord, imbedded in the lower end-strip, falls between the two slots of the clamp. Clamping pressure on the test pieces does not appear to be critical and is applied through wing nuts sufficiently to prevent slippage of the rubber during separation of the cord.

For tests at elevated temperature, 275°F. has been selected for the reasons given in Sec. III, and because it closely approximates the higher temperatures found in tires in service. Twelve test specimens from a particular sample are tightly mounted in twelve separate lower clamps, with cord between slots. These assembled clamps and specimens are then placed in an oven which has previously been brought up to 275°F. Not more than the twelve replicate specimens are heated in an oven for test at one time.

The actual measurement is begun when the specimens have been in the oven for one and a half (1-1/2) hours, by which time, as has been indicated in a preceding section, all specimens have reached a steady temperature of 275°F. Equipped with gloves, the operator quickly removes a lower clamp with its test specimen from the oven and slips it on to the adapter. The free, upper end-strip is brought between the jaws of the upper clamp, the latter is tightened on the test piece, and the lower clamp is set in motion. As soon as the cord has slipped and the reading has been recorded, the upper clamp is opened and the broken specimen and lower clamp are set aside. As quickly as possible another clamp and specimen are removed from the oven and similarly tested. The time required to test ten hot specimens should not exceed fifteen minutes and is usually less. Ten normal separations are regarded as constituting a satisfactory test in routine operations. It is convenient in practice, to keep two ovens in operation, one being used to bring one set of specimens to the required temperature while the other is being emptied and refilled with a new set of specimens.

Adjustment of Results to a "Basic Control"

One especially useful method of discovering serious discrepancies and securing comparability over an extended period of time is through the use of a "basic control" cord sample. This consists of a suitable sample of cord of good uniformity and large enough quantity so that it can be used as an integral part of every test. The use of a "basic control" is especially useful in disclosing variations introduced into the results by a new technician, or the use of a different testing machine, or any other of a number of sources of error.

It has been the practice in these studies to include specimens of cord from the basic control sample in every test. These are treated as though they are one of the experimental samples, are laid in appropriate order in the cord slots of the mold and finally the results are expressed as characteristic adhesion. After a number of separate tests have been run, say ten or more, each containing the basic control cord, an average value is calculated for the characteristic adhesion, K, of the basic control. This is done separately for the tests run at room and at elevated temperatures. In comparing results from different tests, then, the K's for all the samples in a given test are first adjusted by the ratio of the K of the basic control cord in that test to the mean of the K's for all tests, as

$$\text{Adjusted K} = \text{Original K} \times \frac{\text{Average K for Basic Control}}{\text{K for Basic Control of Given Test}} \qquad (9)$$

A control chart may be kept of the characteristic adhesion over the period of test. Such a chart is shown in Figure 11, where the different symbols represent the rubber stocks used. The solid line represents the grand average characteristic adhesion of the control cord, obtained in 41 tests during the first eight months shown on the chart. The dashed lines represent the control limits, calculated from the standard errors of the individual test means according to the procedure recommended by the A.S.T.M. Committee E-1 on Methods of Testing[15]. The limits can, of course, be chosen to meet the particular situation and may be more or less severe, as desired. Then, when a new stock is secured, or some other condition which affects the basic control is changed in such a manner as to increase the variance of the data, or shift the mean adhesion value, this will be made evident by the new values falling outside the control limits more than the expected proportionate number of times.

15/ A.S.T.M. Manual on Presentation of Data, Supplement B - "Control Chart" Method of Analysis and Presentation of Data. See sections headed "Control - No Standard Given", pp. 48, 52-54.

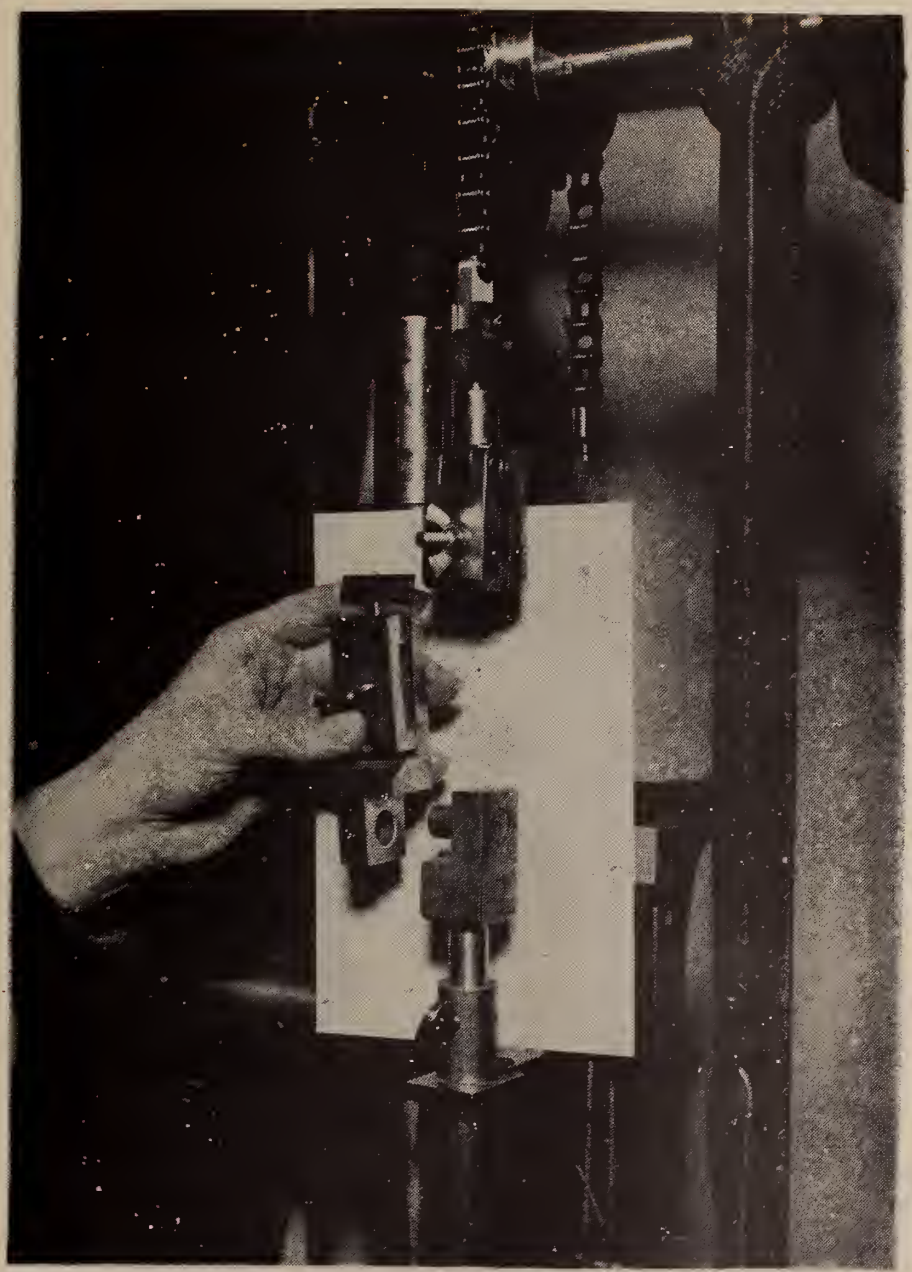

Figure 10. Lower Clamp, with Test Specimen in Place, Being Mounted on Adaptor on Lower Tension Bar of Testing Machine.

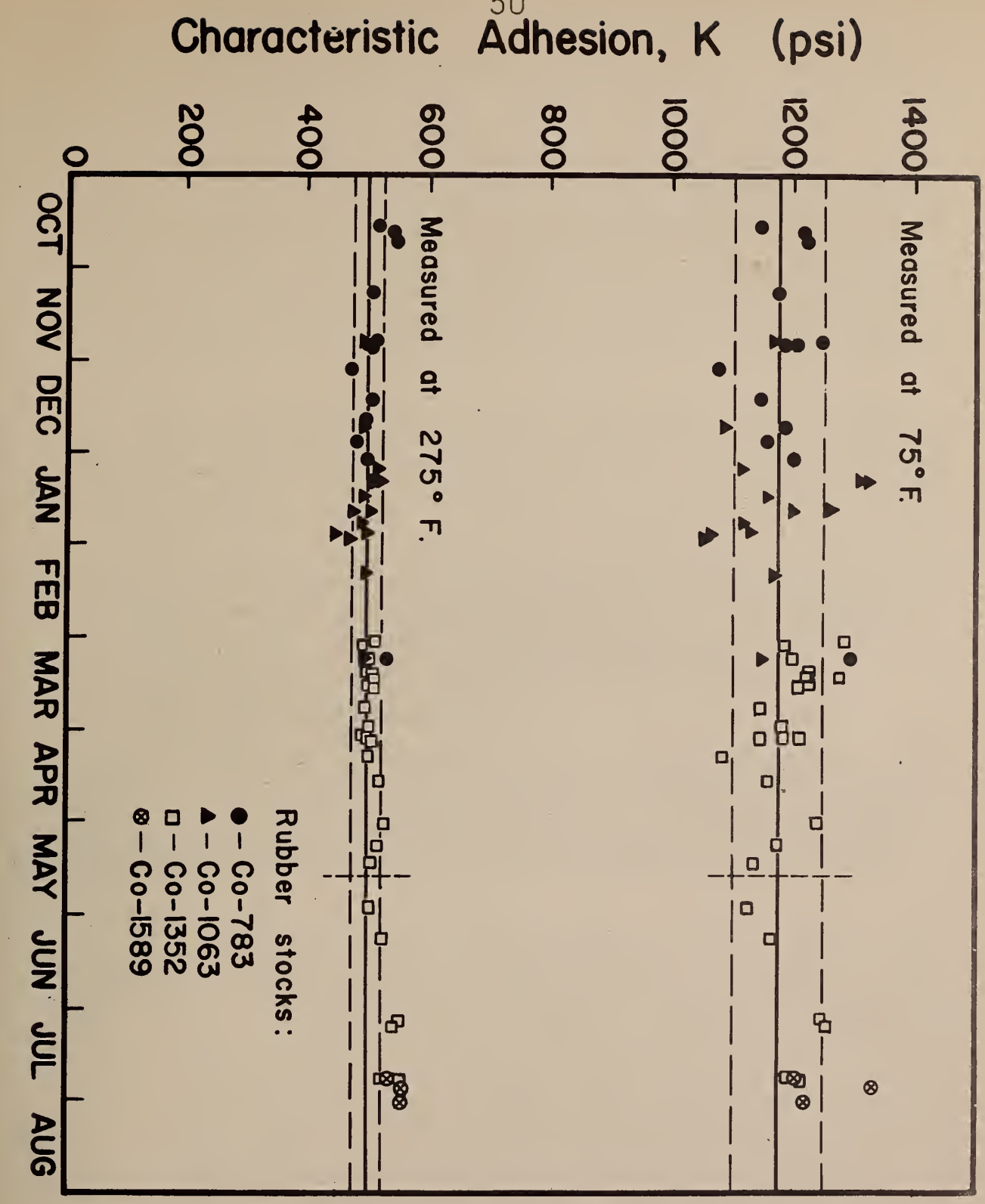

Figure 11. Control Chart for Judging the Homogeneity of Characteristic-Adhesion Means Obtained on the Basic Control Cord. The Central Lines and Control Limits were Computed from the Data of 41 Tests Made During the First Eight Months, Indicated by the Vertical Dashed Lines.

It is obvious from Figure 11 that statistical control was not obtained in some of these tests. For instance, the variability of rubber stocks, indicated in the section on "Rubber-Stock Factors Influencing Adhesion", is confirmed by the greater degree of scattering of mean values for rubber stock Co-1063 than of the other stocks. Several values for this stock fall outside the control limits. The importance of maintaining a standard, uniform rubber stock is emphasized; if this is impossible, a correction must be made to bring the test results to a uniform basis for comparison.

It may also be noted that the values on the chart during the last two months (July and August) do not follow the same trend as the earlier values. This is correlated with the use of a new testing machine, beginning July 1, whose calibration evidently does not agree with that of the old testing machine.

In the present study, as well as in the continuing cord-to-rubber adhesion research in this Laboratory, cotton cord Co-753 has been used as the basic control. From the results on this cord in 41 independent tests, each at 75°F. and at 275°F., average characteristic adhesions of 1175 p.s.i. and 499 p.s.i. have been found, respectively.

Examples of Application of the Method

The application of the H-type adhesion test under the proposed conditions to a series of typical cotton and rayon cords is shown in Table 18. The characteristic adhesions, adjusted to the averages given above for the basic control cord, are shown. Also, at both room and elevated temperatures the adhesions are computed as a percent of the control at that temperature.

By reference to the first section of Table 18 it will be seen that rayon tire cord without treatment has a very low characteristic adhesion, as compared to cotton cord but that the adhesion at both normal and elevated temperatures is nearly up to cotton after treatment with a commercial adhesive. The second section of Table 18 shows typical adhesion results obtained with untreated nylon, rayon, and cotton cords. The characteristic adhesion of nylon is seen to be somewhat better than that of rayon but still far below that of cotton. Slight differences due to use of a new rubber stock are shown in terms of the old stock in the third section of the table. The fourth section of the table shows a series of results obtained on the same control cord after extraction with various solvents, treating with a commercial adhesive and with a solution of polyvinyl acetate. It would seem from this table that a certain assurance is given to the technique as a means for measuring the "adhesion", i.e., the strength of the cord-to-rubber bond.

Precision of the Method

Since, as has just been stated, the basic control cord was included with every series of cord samples on which adhesion measurements were to be made, the results obtained permit the standard error of the method to be calculated. The results are shown in Table 19 for both normal and high temperature measurements. The results may be compared with the average standard errors predicted from the within-test variability. Table 19 also shows the standard error of means of 10 observations each, expressed as a percentage of the mean characteristic adhesion. It will be seen that the overall errors based on the use of the same cord in all tests are very considerably larger than would be predicted from the within-test variability. This analysis shows, therefore, the presence of further still unidentified and uncontrolled variables. At the present time the precision of this technique at normal temperatures, in terms of standard error, may be taken as approximately 5 percent, and at 275°F. as approximately 6 percent.

Table 19. Observed and Computed Standard Errors of Means of 10 Observations Each.

Source of standard errors	Characteristic Adhesion	
	At 75°F.	At 275°F.
	Percent	Percent
Based on 41 different tests	4.9	5.9
Computed from within-test variations	2.6	3.2

IX. SUMMARY

While there are several types of tests in current use for measuring the adhesion of fabric and cords to rubber, the "H" adhesion test has been adopted tentatively at the Southern Regional Research Laboratory for evaluating the adhesive properties of cotton and other tire cords in natural and GR-S rubber. Essentially, the "H" test may be said to be a measurement of the force required to pull a cord, in the direction of its length, from a strip of rubber held in a clamp. Since very little information on the characteristics and reliability of the "H" test appeared to be available, the present study was undertaken. The various factors which could have been expected a priori to influence the adhesion or its measurement, fall into four broad classes: experimental conditions, time factors, rubber-stock factors, and cord factors.

Table 18. Application of "H" Adhesion Method to Various Tire Cords.

Sample No.	Tire cord and treatment	Cord gage	Characteristic Adhesion[1]			
			At 75° F.		At 275° F.	
			Absolute	Relative[2]	Absolute	Relative[2]
		Ins.	p.s.i.	Percent	p.s.i.	Percent
Co-973	Rayon cord, untreated, 1100/2	0.0240	275	100	188	100
Co-1157	" " , treated commercial adhesive	.0255	969	352	435	231
Co-971	Nylon cord 210/4/2	.0160	307	-	212	-
Co-893	Rayon cord, untreated	.0224	235	-	162	-
Co-968	Cotton cord, Standard 16/4/3	.0342	1119	-	471	-
Co-753	Cotton cord, GR-S Stock Co-783	.0315	1175	100	499	100
Co-753	" " , initial test of new GR-S Stock Co-1063	.0315	1077	92	496	99
Co-753	Untreated	.0315	1175	100	499	100
Co-1233	Co-753 extracted ethyl acetate	.0325	1226	104	498	100
Co-1234	Co-753 " acetone	.0314	1211	103	509	102
Co-996	Co-753 " benzene	.0330	1176	100	515	103
Co-1444	Co-753 " xylene	.0288	1130	96	433	87
Co-1061	Co-753 with commercial adhesive	.0364	3/	3/	633	127
Co-1232	Co-753 with polyvinyl acetate	.0337	710	60	354	71

1/ Adjusted to mean of basic control cord.

2/ Based on untreated cord.

3/ Cord broke before separation occurred.

The pre-drying of cotton cord, and variations in the pressure on the mold over the range 300 to 700 p.s.i., were found to have no significant influence on the adhesion measurements. As was anticipated from the behavior of cords in tires in service, the adhesion is found to be greatly dependent on the temperature prevailing at the time of measurement. It has been shown that the force of adhesion of a typical untreated cotton cord, both to natural and GR-S rubber, when tested at 275°F., drops to about 40 percent of its value at room temperature (75°F.). A rather poor correlation was found between the adhesion at room temperature and that at 275°F., of a wide variety of cords, leading to the conclusion that actual, high-temperature measurements are a necessary part of a routine testing procedure.

Among the factors involved during the molding stage of the test operations, the direction of calendering of the raw rubber stock, and differences in the types of molds used, were found to have, in general, little or no influence on the results. The position of the specimen in the mold during molding appears to have no important influence on the magnitude of adhesion measured on that specimen. Any small influence that may prevail, can be largely offset by the arrangement of the different samples of cord in progressive succession in the cord slots of the mold.

The rubber sheath around the cord between the end strips of each H-type test-piece was found to cause significant but spurious increases in the adhesion value at both room temperature and 275°F. In some cases these increases exceeded 25 percent of the total force; the influence of the sheath on the test-piece during actual test is regarded as one of the most important sources of error encountered.

Two factors associated with the testing machine were investigated: the speed of the pulling clamp, and the conditions under which the test specimen is clamped. It was found that increases in clamp speed produced significant increases in the observed adhesive force at room and at elevated temperatures. For the standard, uniform speed which these results dictate, 12 inches per minute is recommended. No real distinction is apparent between the observations obtained with deep serrated jaw-surfaces and those obtained with knurled surfaces, nor does it appear that the pressure with which the specimens are clamped, can sensibly affect the observations.

Adhesion, measured at room temperature, was found to increase slightly during the first 24 hours after cure. Within this period a stable value is attained, which is retained for at least a month. Adhesion measurements at 275°F. are not changed by the period of delay between curing and testing. Thermocouple measurements indicate that 1-1/2 hours is an adequate heating period in which to bring specimens up to a testing temperature of 275°F.; one hour appears to be too short a period. If heated specimens are allowed to cool back to room temperature before testing, the adhesion returns to the value observed before heating, indicating that no permanent chemical or structural change occurs in the rubber or cord as a result of the heating. If the test is made within one minute of removal of the specimen from the oven, the cooling is too brief to have a measurable effect on the adhesion.

Tested at 75°F., as well as at 275°F. specimens containing natural rubber gave adhesive forces which were approximately equal to those for specimens containing GR-S; however, at intermediate temperatures the adhesion of cotton cord to GR-S rubber was inferior to that of the natural rubber stock of the type used in the present tests. Different batches of GR-S stock are found to behave somewhat differently, also. However, due perhaps to judicious blending, in the rubber stocks studied thus far, these differences are suppressed and negligible in the 75°F. and 275°F. tests. The adhesive forces measured on specimens containing the same GR-S rubber and cord samples, but molded many weeks apart, are found to remain substantially uniform at the various test temperatures when the uncured rubber stock is stored in a refrigerator at approximately 45°F. However, when the rubber supply is stored at room temperature for long periods a drop in adhesion measured at room temperature results. Tests made on a representative roll of GR-S stock indicate that the adhesive forces associated with different areas of the single roll are uniform within the limits of experimental error.

The force required to separate a cord from rubber was found to increase linearly with the gage. For an equitable comparison between cords of different gages, an adjustment must be made for the difference in gage. The "characteristic adhesion", which has been introduced and defined as the adhesion referred to a cord fabric one inch square, provides a quantity in terms of which the adhesion results may be expressed independently of gage. As has been shown, the characteristic adhesion may be used to obtain a practical estimate of the adhesion prevailing between unit areas of rubber and tire fabric, the latter of which may be composed of any one of the various types of cords, or may vary in the numbers of cords per inch of width.

No distinction in adhesion could be drawn from the results on 2- and 3-ply cords, on the basis of the number of plies in a cord. The twist of a cord, however, was found to have a small influence, the characteristic adhesion increasing with the number of turns per inch. Adjustment formulae have been presented to permit the comparison of characteristic adhesions of cords having different twists.

While a rigorous comparison of the effects of the various physical factors entering the "H" test is not possible, the four important factors might be ranked as follows: (1) testing temperature, (2) presence of the sheath around the cord, (3) gage of the cord, and (4) speed of the pulling clamp. The other factors have not been found to be critically relevant, within the ranges of sampling covered by this study. The use of adhesives on cotton and rayon cord markedly influences the adhesion, of course, but such effects are considered to be outside the scope of the present paper.

A description of a recommended procedure for conducting the "H" adhesion test is given. The proposed method, based on the findings of the present study, aims to provide means for evaluating or holding uniform those factors which have a significant influence on the results. The manner in which a selected sample of cord may be used as a control, for the adjustment of the results of individual tests to a common basis, is shown. Data accumulated during the study reveal that the standard error in the characteristic adhesion obtained by use of the proposed method, may be expected to be about 5 percent at normal temperatures and about 6 percent at 275°F.

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